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Proceedings of the First Annual Symposium for Nondestructive Evaluation of Bond Strength

Compiled by
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PREFACE

Thirteen nondestructive evaluation (NDE) experts met for the First Annual Review of NASA's NDE of Bond Strength Program at LaRC, NDE Sciences Branch on November 4, 1997. The goal of this research is to nondestructively determine quantitative strength levels in structural bonds. The Symposium was held to review both "in house" NDE research and work performed by sponsored university grantees. The grants reviewed were: "Nondestructive Determination of Bond Strength", The Johns Hopkins University (Dr. Robert E. Green and Mr. Tobias P. Berndt); "An Ultrasonic Technique to Determine the Residual Strength of Adhesive Bonds", Northwestern University (Dr. Jan D. Achenbach and Mr. Zhenzeng Tang); "Ultrasonic Nondestructive Characterization of Adhesive Bonds", The Georgia Institute of Technology (Dr. Jianmin Qu and Mr. Larry Jacobs). An invited presentation, "Preliminary Attempts to Detect Weakness of Adhesive Bonds", was given by Dr. Donald Price of the Computational Industrial Research Organization (CSIRO, Sydney, Australia). Several technologies and approaches were presented including "Adhesive Model with Varying Interfacial Layers Using Longitudinal Ultrasound", by Mr. Robert Anastasi and Dr. Mark J. Roberts, ARMY-VTC, "Surface Contamination Monitoring using Optically Simulated Electron Emission (OSEE)", Dr. Christopher S. Welch, College of William and Mary. Nonlinear ultrasonics is being investigated as a possible lead technology for nondestructively detemining bond strength. The Symposium proceedings are published in this NASA Conference Publication.

Bond Strength Program

Dr. Mark J. Roberts

Tuesday, 4 November 1997

Schedule

NDE BRANCH WELCOME

8:30-8:45 Dr. Edward Generazio (Head)

NASA BOND STRENGTH

8:45-9:00 Program Overview & In House Efforts : Dr. Mark Roberts

9:00-9:10 Adhesive Interfacial Layer Modeling : Dr. Mark Roberts

9:10-9:40 Surface Contamination Characterization: Dr. Chris Welch

9:40-9:50 Ultrasonic System (SUSAN): Dr. Patrick Johnston

GRANTEE PRESENTATIONS

9:50-10:20 Johns Hopkins University: Dr. Bob Green

10:20-10:35 Break

10:35-11:05 Georgia Tech: Dr. Jianmin Qu

11:05-11:35 Northwestern University: Dr. Jan Achenbach

LUNCH 11:35-1:00

GENERAL SESSION

1:00-1:30 NDT of Bonded Structures, CSIRO: Dr. Donald Price

1:30-3:00 General Discussion Period

3:00 ADJOURN

Overview & In House Research

- Program Issues
- Goals & General Approach
- Justification & Needs
- Resources & Collaboration
- In House Research

Program Issues

- Customer Needs & Requirements
  - Aircraft operational safety
  - General structural integrity (Composite Structures)
- Nondestructive Evaluation Methods
  - Ultrasonics
  - Microwaves
  - Thermal
  - SAM
- Mechanical Testing Methods / Destructive
  - Lap Shear
  - Fatigue test
  - Loading tests: static, dynamic

Program Issues

- Bondline Strength Concerns and relation to
  - Surface Preparations
  - Quality Control
  - Cure State
  - Environmental Conditions
  - Durability
  - Interfacial Conditions

- Results of Successful Program
  - Could eliminate or reduce rivet / fastening technology
  - Production of higher strength bonds
  - Measurement technique to quantitatively measure strength
  - Reduce operational downtime of aircraft significantly

BOND STRENGTH PROGRAM

- MAIN GOALS
  - Develop clear understanding of bond strength in general structures
  - Develop NDE methods for measuring bond strength & bond quality levels
  - Develop prototype system and specifications for bond strength NDE analysis

- APPROACH
  - State of the art review is an "ongoing process"
  - Industry / NASA / University Teams consolidating
  - Select NDE technologies best suited for the bond strength problem solution
  - Perform strength measurements using various NDE methods
  - Begin system prototype design of bond strength instrument

- JUSTIFICATION & NEEDS
  - Fulfills FAA safety requirements for bond manufacturing
  - Cost reduction & less operational down time possible using prototype measurement system
  - Adhesively bonded structures cheaper to produce
  - HSR Systems will utilize bonded structures

- RESOURCES
  - $800K over 5 years, projected to 2002
  - Civil Servants : 4 NASA (projected), 1 ARL
  - NASA LaRC NDE Laboratories & University Centers for NDE (Johns Hopkins, Northwestern University, Georgia Tech)
  - Boeing Aircraft, Seattle, WA (Collaborative)
  - CSIRO Sydney, Australia (Collaborative)
  - Colorado State University

BONDING KEY ISSUES

- Defining adhesive bond strength
- Stiffness versus strength of adhesive layer
- Surface Preparation / Bond Cure State
- Quality Control in Manufacturing Process
- Fracture Properties of Adhesive
- Bond Durability
- Environmental Effects & Contamination
- Moisture / Water in the Bondline
- Interfacial Boundary Properties
- Existence of Weak Boundary Layers
- Mechanical interlocking
- Microstructural Properties

In House Research

- Fiscal Year Goals of In House Resarch
- Bond Research Topics

Bond Strength GOALS : FY97-FY00

- FY97
  - Reviewed existing *state of the art* technology relevant to bond strength evaluation
  - Clearly define all bond issues related to bond strength problems
  - Examine various NDE testing methods to address quantitative bond analysis
  - Conducted in-house feasibility to use microwave and laser ultrasonic methods
  - Theoretical discussions: Colorado State's AMNTL
  - University research grant work for NASA underway for 1 year plus

Measurement Study FY98

- Experimentally measure strength of simply adhesively bonded structures by means of destructive methods
  - fatigue & peel testing
  - fracture testing
  - time durability measurements
- Establish physical understanding of strengths being measured both destructively and non-destructively
- Correlate bond strength to experimental measurements
- Compare numerical and analytical methods in bond evaluation analysis

- Experimentally measure strength of simply adhesively bonded structures by means of NDE methods
  - Nonlinear Ultrasonics
  - Prestressed small signal ultrasonics
  - Thermal methods
  - Microwave moisture measurements
- Use recommendations of university research PIs in year 2 to choose best NDE methods for continued measurement, modeling & analysis.
- Narrowing down of possible technologies to a select few which produce a direct measurement correlation with bond quality and strength

Bond Strength FY98

- Study bondline failures caused by crack propagation in structure due to microstructural changes

- Investigate the concept of interfacial and weak boundary layers occurring at the adhesive - adherend boundary and its effects on bond strength

- Computer simulate the interfacial / weak boundary layer model for an adhesively bonded structure

- Develop clear understanding of bond durability and how it is affected by surface prep, environment (moisture, water, heat)

Bond Strength FY99 - FY02

- FY99
  - Finalize approach to problem solution to prepare for analysis of more generic adhesively bonded structures
  - Apply knowledge to directly measure bond strength in any generic structure, i.e., composite aircraft
- FY00
  - Begin design of benchmark bond measurement system based on modeling & NDE experimentation
- FY01
  - Begin development of a final prototype instrument
- FY02
  - Final bond strength measurement instrument projected completion - Sept. 2002

In House Research

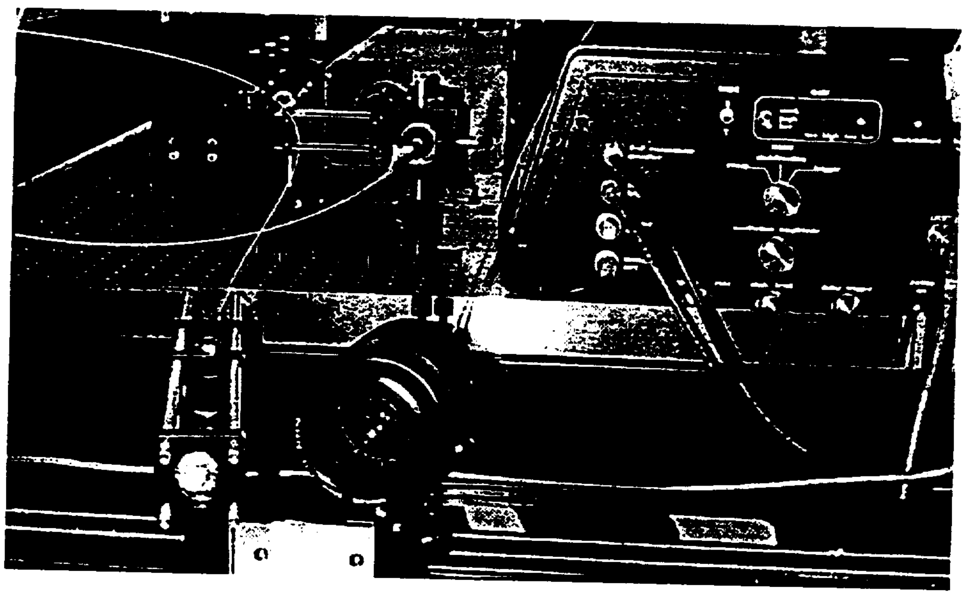
- Bond Research
  - High Speed Research (HSR) Program Issues
    - Bond Importance
      - Bonded structures used extensively in HSCT
      - No use of back up fasteners necessitates bond assessment
    - Bond Issues
      - Composite / Composite Co-cured bonds
      - Composite / Metal Bonds
  - Computational Chemistry Approaches to Bondline Analysis
  - Theoretical chemical bond strength of carbon fiber-polymer matrix interfaces

In House Research (cont.)

- Bond Research (cont.)
  - Laser Ultrasonic System to be set up soon for analyzing adhesively bonded specimens
  - Microwave Methods - Collaboration with Colo. State Univ.
  - Optically Stimulated Electron Emission (Wm. & Mary)
  - SUSAN - Ultrasonics System
  - Adhesive Model with Varying Interfacial Layers using Longitudinal Ultrasound

In House Research

- Investigate a non-contacting approach.
  - Laser Ultrasonic System
    (Under construction.)



- Introduce large amplitude strains
  - Mechanically or Thermally
- Utilize Laser UT System to try to probe the bonding layer while under stress to measure components of the higher order elastic constants.

Computational Investigations of Adhesive Bonds

- Goals
  - Determine molecular level mechanisms for bond deterioration
  - Determine quantitative characteristics of molecular level structures in good and deteriorated bond structures

- Approach
  - Use computational chemistry methods (first principals an-initio and local density approximation; semi-empirical; and molecular modeling methods
  - Start with small models of chemical structures at interfaces and determine properties of molecular bonds (stiffness - i.e harmonic force constants, and nonlinear properties of chemical bonds- i.e. anharmonic bond force constants)

Computational Investigation of Adhesive Bonds (continued)

- Approach (continued)
  - Repeat investigations after adding H2O to model structure
  - Iterate with more comprehensive model structures and other environmental changes(addition of Cl atoms, oxygen, etc.)

- Implementation
  - Hardware: Modern Workstation with large memory and disk space
  - Software: Intitally use available codes (Gamess, DeFT, Moil, etc.) progress to commercial software if needed

Future In House Steps

- Continue literature research as more new information becomes available
- Build on existing results provided by university grantees
- Be open to technologies not previously used which could provide scientific insight into bond strength solution
  - Microwave
  - SAM
  - Optics
- Provide laboratory experimentation ultrasonically on peel ply specimens which represent "weakened" bonds provided by Boeing Aircraft
- Examine mathematical modeling possibilities

Adhesive Model with Varying Interfacial Layers Using Longitudinal Ultrasound

4 November 1997

Robert Anastasi

Mark Roberts

Table 2. CASE 2 - ANALYTICAL PARAMETER LIST

	Velocity (v) (m/s)	Density (ρ) (kg/m^3)	Impedance (z) (x10^6 kg/m^2 sec)	Thickness (d) (m)
Adherend	6370	2710	17.25	Semi-infinite
Adhesive	2100	1120	2.35	70.0×10^{-6}
Interface Layers:*				
Model-a 100%	2100	1120	2.35	7.0×10^{-6}
Model-b 50%	2100	560	1.18	7.0×10^{-6}
Model-c 20%	2100	220	0.47	7.0×10^{-6}
Model-d 10%	2100	110	0.23	7.0×10^{-6}

*Percentage of adhesive density, corresponding impedance calculated using $z = \rho v$

Table 3. CASE 1 - NUMERICAL PARAMETER LIST

Thickness (m)	V_L (m/s)	V_S (m/s)	#Z Elements	$\lambda_L/8$ (m)	$\lambda_S/8$ (m)	Δt (nsec)	ΔZ (m)	$\Delta Y/\Delta Z$
3.17×10^{-3}	6370	3110	123	5.3×10^{-5}	2.59×10^{-5}	2.861	2.58×10^{-5}	0.998
7.00×10^{-6}	6370	3110	1	5.3×10^{-5}	2.59×10^{-5}	1.060	7.00×10^{-6}	3.68
7.00×10^{-5}	2100	1050	9	1.75×10^{-5}	8.75×10^{-6}	3.542	7.77×10^{-6}	3.31
7.00×10^{-6}	6370	3110	1	5.3×10^{-5}	2.59×10^{-5}	1.060	7.00×10^{-6}	3.68
4.77×10^{-3}	6370	3110	185	5.3×10^{-5}	2.59×10^{-5}	2.861	2.58×10^{-5}	0.998

#DOF = 316160 nz = 320 ny = 494 Δt = 1.060 nsec

Table 4. CASE 2 - NUMERICAL PARAMETER LIST

Thickness (m)	V_L (m/s)	V_S (m/s)	#Z Elements	$\lambda_L/8$ (m)	$\lambda_S/8$ (m)	Δt (nsec)	ΔZ (m)	$\Delta Y/\Delta Z$
3.17×10^{-3}	6370	3110	82	7.96×10^{-5}	3.88×10^{-5}	4.296	3.87×10^{-5}	1.000
7.00×10^{-6}	2100	1050	1	2.625×10^{-5}	1.313×10^{-5}	3.280	7.00×10^{-6}	5.531
7.00×10^{-5}	2100	1050	6	2.625×10^{-5}	1.313×10^{-6}	5.316	1.166×10^{-6}	3.319
7.00×10^{-6}	2100	1050	1	2.625×10^{-5}	1.313×10^{-5}	3.280	7.00×10^{-6}	5.531
4.77×10^{-3}	6370	3110	123	7.96×10^{-5}	3.88×10^{-5}	4.304	3.882×10^{-5}	0.9974

#DOF = 140812 nz = 241 ny = 329 Δt = 3.280 nsec

Table 5. FREQUENCY MINIMA COMPARISON

Case/Model		Density of Interface Layers (kg/m^3)	Frequency Minima Locations: Analytical (MHz)	Frequency Minima Locations: Numerical (MHz)
Case 1	Model-a	2710	15.00	14.93
	Model-b	1360	14.80	14.73
	Model-c	540	14.35	14.31
	Model-d	270	13.75	13.72
Case 2	Model-a	1120	12.50	12.40
	Model-b	560	10.80	10.80
	Model-c	220	8.20	8.12
	Model-d	110	6.20	6.16

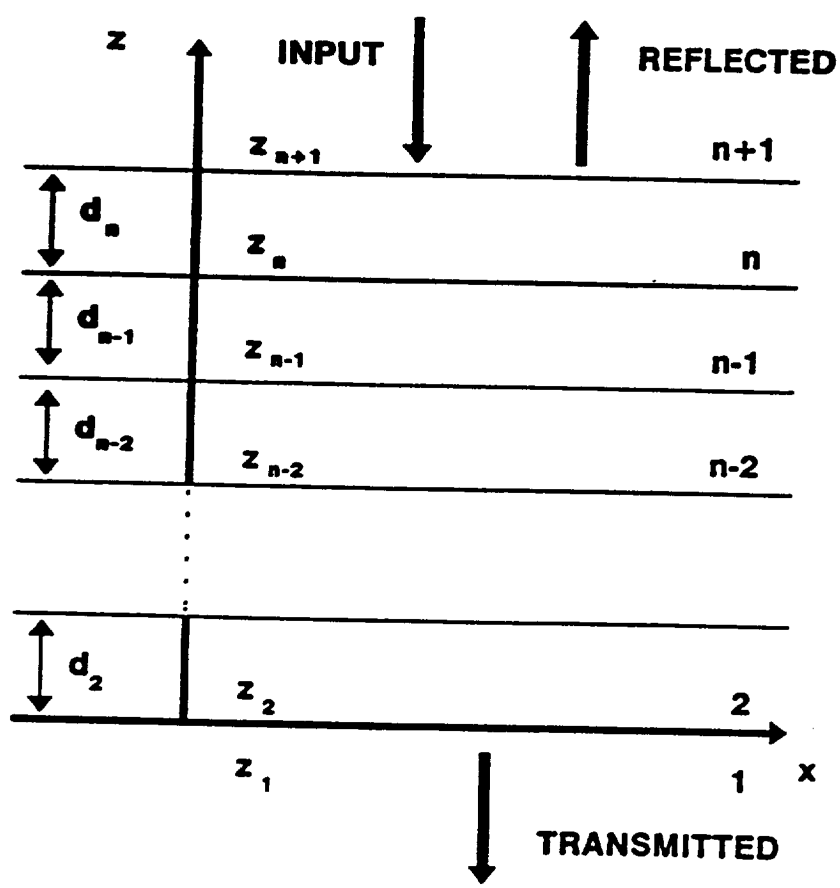


Figure 1. Multi-layered structure model used for calculation of reflection coefficient.

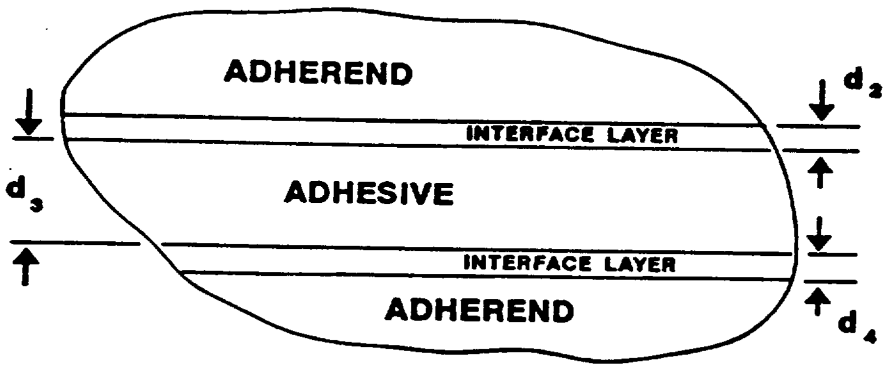
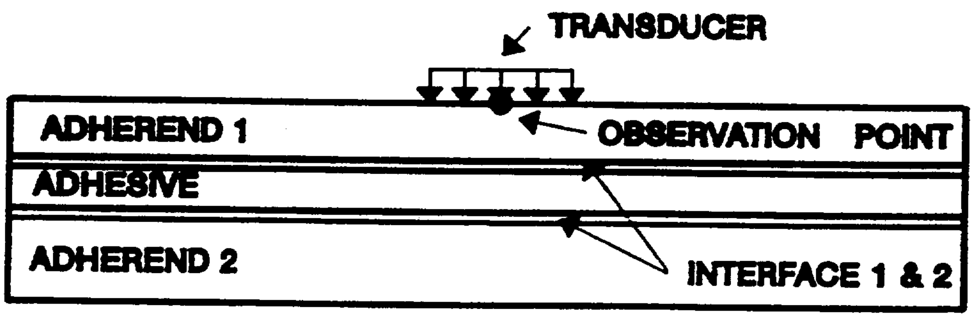


Figure 2. Analytical representation of adhesively bonded structure.



LAYER	THICKNESS
ADHEREND 1	3175 μm
INTERFACE 1	7 μm
ADHESIVE	70 μm
INTERFACE 2	7 μm
ADHEREND 2	4775 μm

Figure 3. Adhesively bonded structure geometry with finite aperture transducer pulsed excitation (adherends are aluminum).

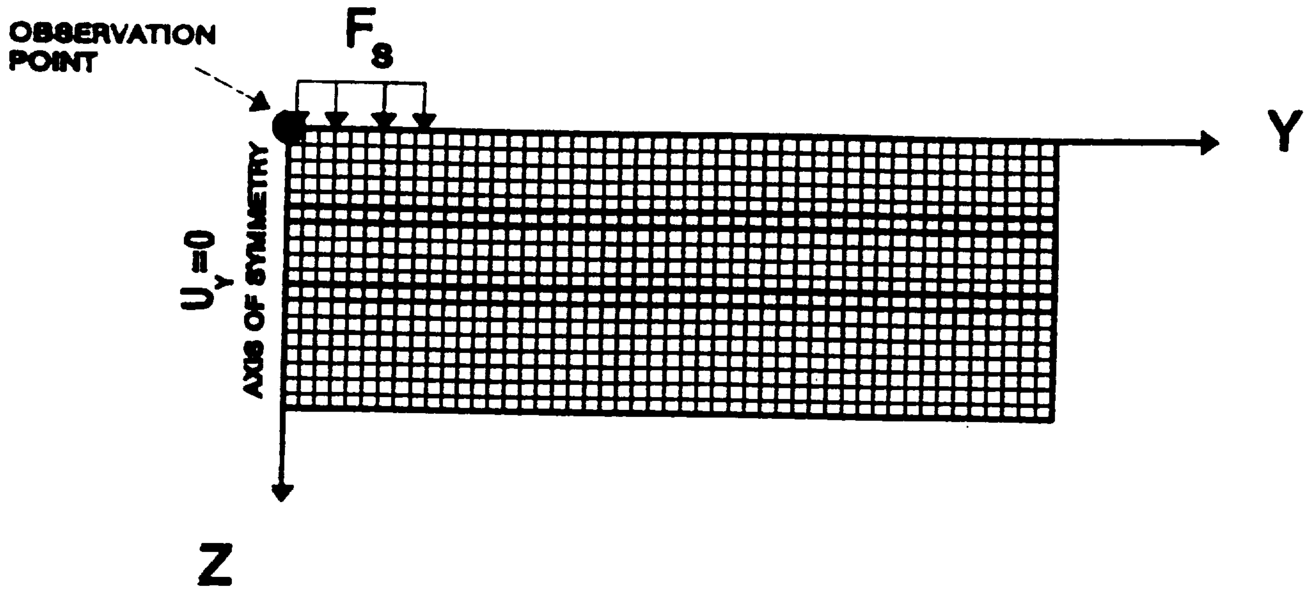


Figure 4. Possible finite element mesh of five-layered structure exploiting mechanical symmetry, shear displacements set to zero-value on symmetry axis.

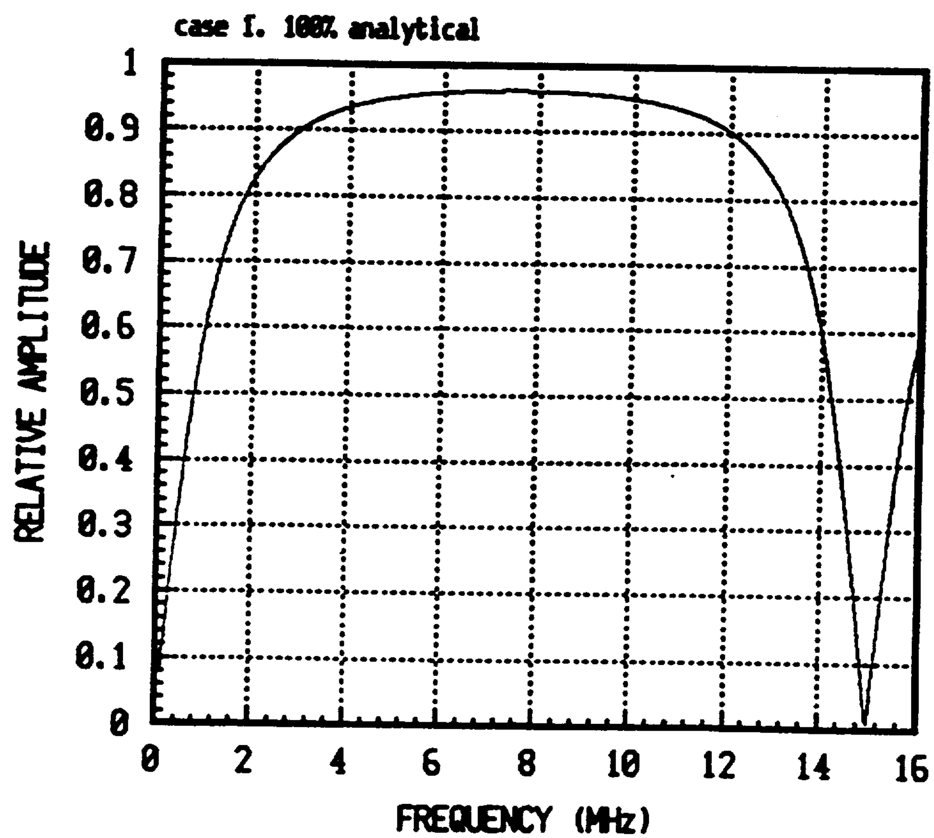


Figure 5. Analytical reflection coefficient Case 1 (good bond).

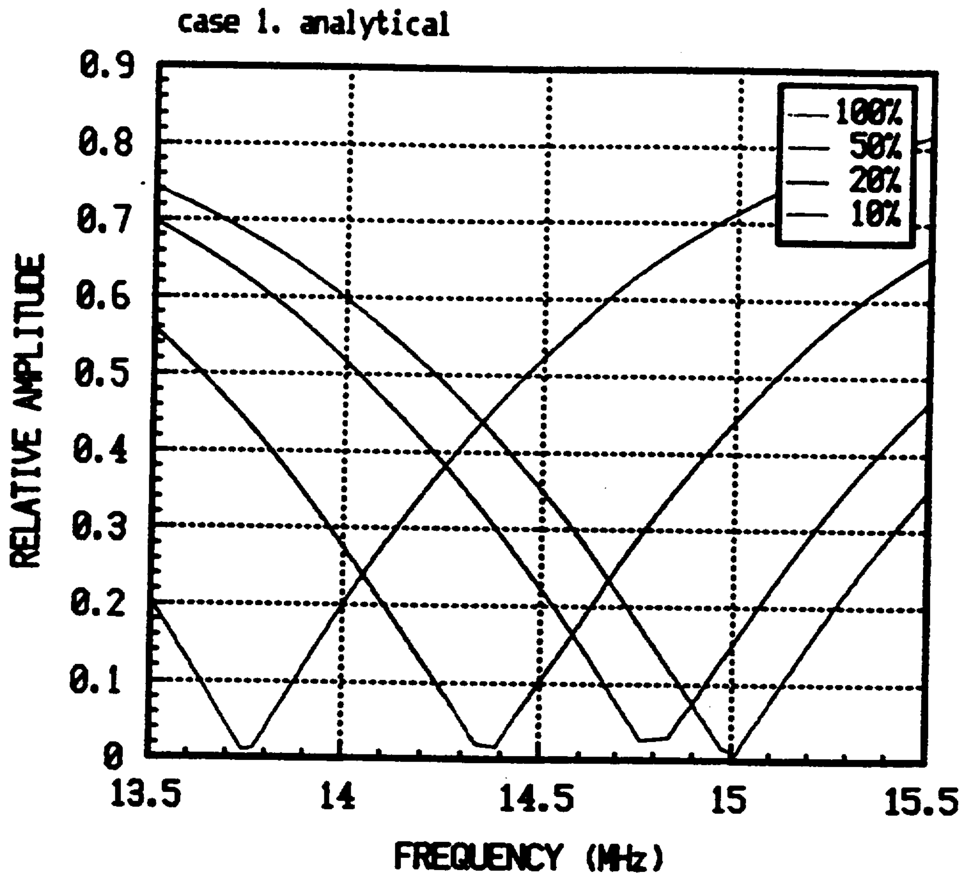


Figure 6. Analytical reflection coefficients for Case 1. Bond quality levels: 10%, 20%, 50%, 100% - 13.5 ≤ f ≤ 15.5 MHz.

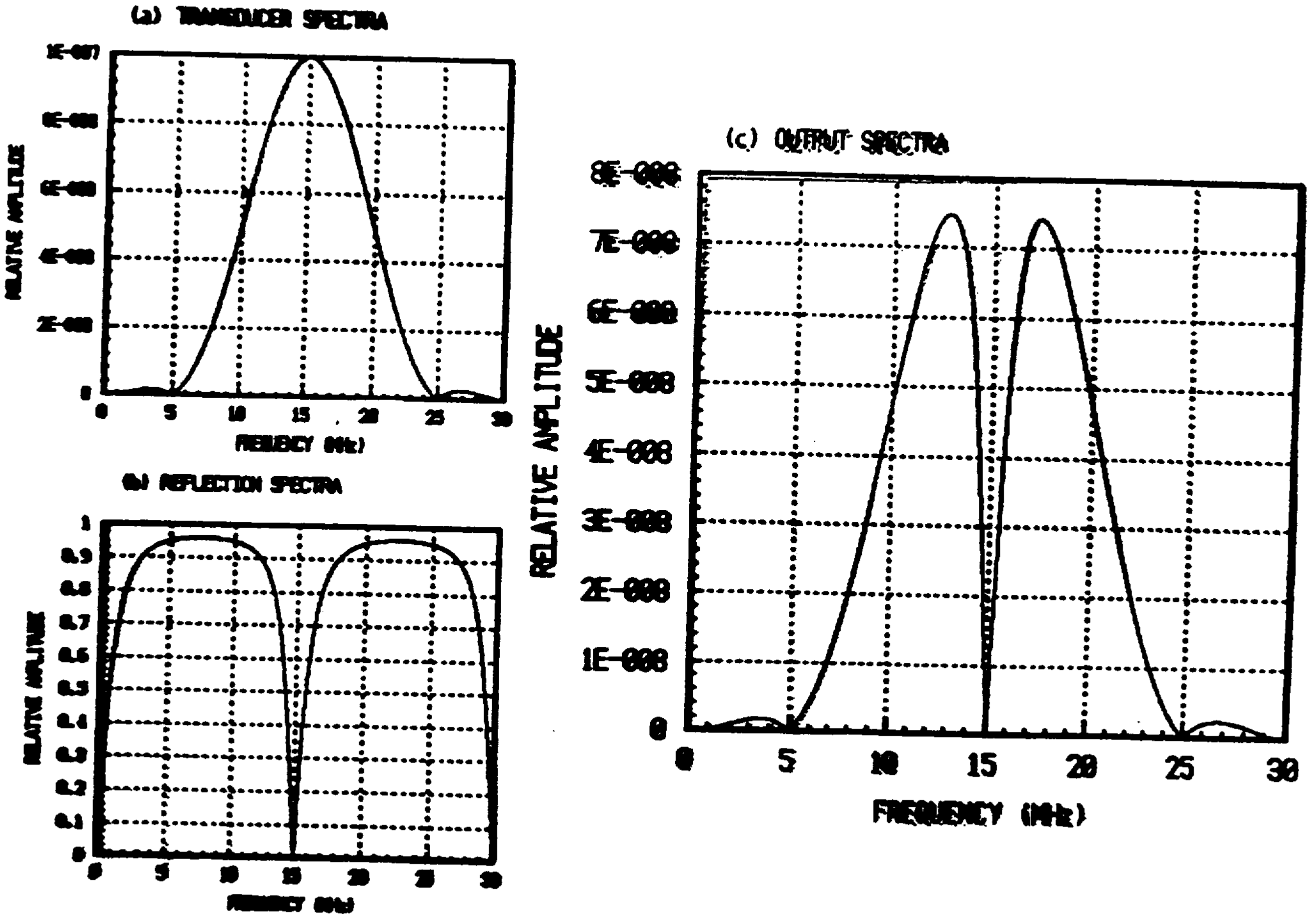


Figure 7. Analytical frequency domain analysis for good bond (100%), Case 1: (a) Fourier transform of raised cosine with $f_0 = 15$ MHz, (b) Reflection coefficient of adhesively bonded model, and (c) Relative amplitude of transformed mechanical displacement response.

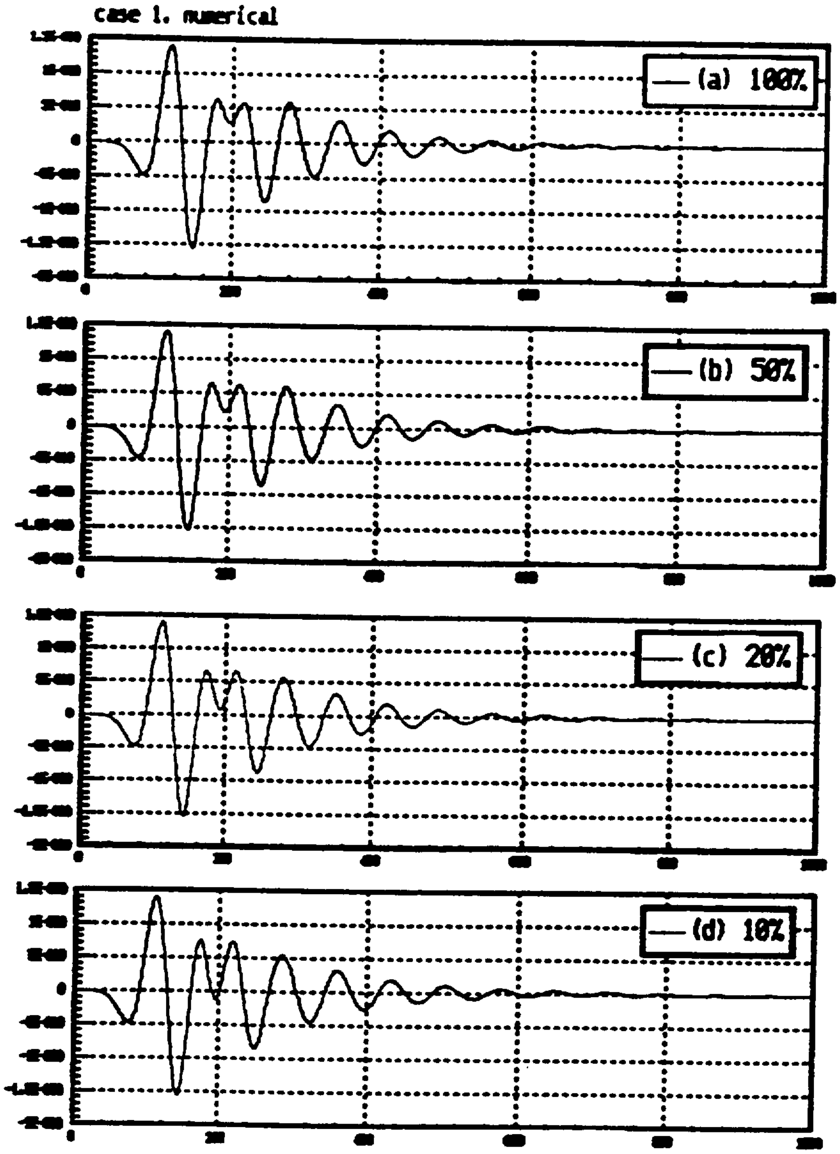


Figure 8. Time domain mechanical displacement responses, Case 1: (a) 100% bond quality level, (b) 50% bond quality level, (c) 20% bond quality level, and (d) 10% bond quality level.

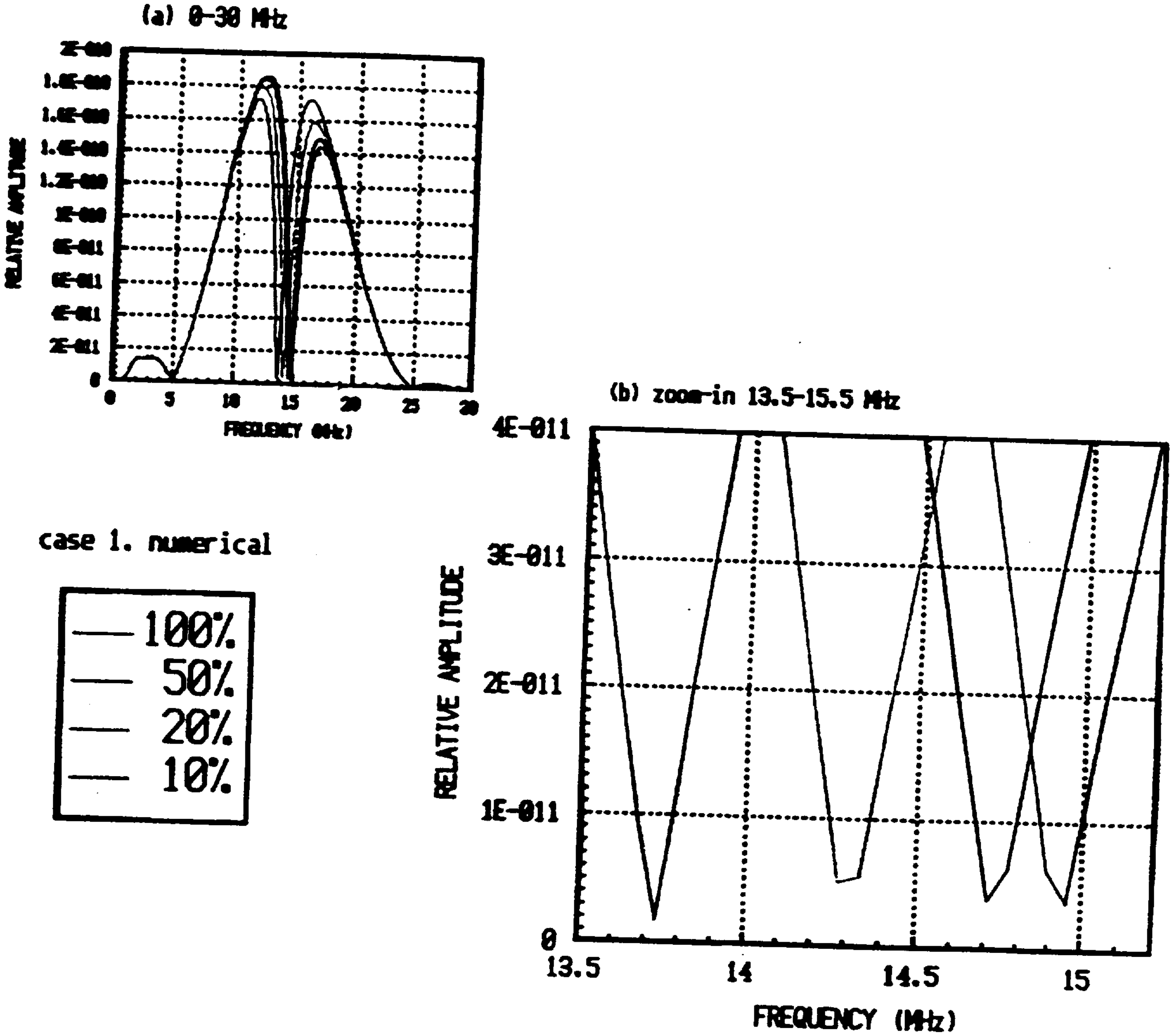


Figure 9. Frequency domain mechanical displacement responses, Case 1: (a) Responses for frequency range $0 \leq f \leq 30$ MHz, and (b) Responses for frequency range $13.5 \leq f \leq 15$ MHz.

Optically Stimulated Electron Emission (OSEE) for Bond Inspection

Christopher S. Welch
Applied Science Department
College of William and Mary

1. Description of OSEE

Optically Stimulated Electron Emission (OSEE) is a non-destructive inspection technique which has been developed by NASA and its contractors to verify the cleanliness of bonding surfaces. With OSEE, verification occurs immediately prior to applying adhesive and forming the bond. OSEE was developed to address the realization that a major cause of failure in bonded joints is contamination of the bonding surfaces prior to bond formation.

The technical basis for OSEE is that the efficiency of the process of photoelectron production by ultraviolet light is highly sensitive to the state of the emitting surface, so that small amounts of contamination can greatly change the photocurrent produced by a given amount of light. It was found that the charges corresponding to the emitted photoelectrons could be attracted to a positively charged anode, even through a considerable amount of ambient air. This discovery permitted design of non-destructive instrument (shown schematically in Fig. 1) to inspect bonding surfaces in the manufacturing setting. In Fig. 1, an anode, biasing battery, ammeter and circuit ground connection are added to the photoelectron-emitting surface. The resulting complete circuit is the basis of the measuring instrument, with the current measured by the ammeter becoming the measurement.

2. OSEE and Bonds - A Brief History at NASA

In the early days of the NASA Space Shuttle, it was recognized that the bond between the solid rocket booster case and its insulation/fuel package was critical to the operation of the motor, and that a failure in this bond during operation could easily lead to a burn through the case and a possible mission failure. This realization drove a need to inspect the bonding surfaces prior to the application of the first layer of insulating material to the steel of the case. This need was particularly urgent with refurbished motors. Small residual amounts of the rust-preventative used on the motors following recovery from the ocean were shown to weaken the bondline, if they remained after cleaning (Gause, 1989). Inspection of the bonding surface, which was grit-blasted after degreasing, was difficult, and optical methods of inspection designed for smooth surfaces were not applicable, because the surface was not smooth, but grit-blasted. A scientist working in the research laboratory of the prime contractor (Smith, 1979) recognized the potential for OSEE to address the inspection, and the idea was put into development (Smith, 1986) and deployment, with a commercial firm coming forward to build and supply the equipment. In rapid order, an instrument was designed, configured, and put into service inspecting Shuttle solid rocket motor casings.

After some time, field experience with the OSEE instrument brought out a need for some improvements. It became evident that the commercial firm which was supplying the instruments, a small business with little other commercial base, lacked the research infrastructure to undertake an extensive investigation into the factors which produced variations in the OSEE readings. The instrument was important to the NASA mission, so the task of investigating the factors was assigned to the NDE laboratory at NASA Langley Research Center. This investigation became known as the OSEE science base study.

3. Findings of the science base study

The science base study identified several factors which affect OSEE readings as well as putting into perspective the factors governing OSEE operations. To show that variability is not an intrinsic part of OSEE measurements, an effort was made to reduce variability. This effort eventually achieved reproducibility within 1 percent of the OSEE current in two measurements on a clean surface over time (Fig. 2). The biggest factor in attaining reproducibility was the use of an argon purge to reduce photochemistry in the measurement region (Welch, *et al.*, 1992). Reproducibility led to the ability to perform comparative experiments for factors which might produce variability. These comparative experiments produced several findings of significance. It was determined that the only portions of the lamp spectrum (a low-pressure mercury lamp) which produced significant photocurrent were the 185 nm line and the 254 nm line, the 185 nm line producing about 95% of the total current. OSEE variations on clean surfaces were found to be sensitive to variations in the work function of the surface. Sensitivity was found to even trace amounts of humidity in the atmosphere surrounding the measurement, and to small variations in the temperature of the lamp envelope. Also, the voltage-current characteristic of the OSEE process was found and related to early work in gaseous electronics. A verification of the sensitivity to contamination was done, and a sample cleaning technique developed (Abedin,*et al.*, 1992).

4. Dielectric substrates

In a follow-on to the science base study, a procedure was developed which achieved reproducible OSEE data on a nonconducting substrate. This procedure, named charge replacement, led in part to a patent (Yost, *et al*, 1995), because it opened the opportunity for OSEE to inspect all surfaces, not just metal surfaces (Welch and Yost, 1995). This ability permitted performance of a study of the applicability of OSEE to inspect surfaces of electronic assemblies for residual solder flux in various assembly processes under study by the electronics production industry (Welch, 1995).

5. OSEE Instrumentation Development

With the improved understanding of the operation of OSEE, authority was extended to design and build an improved OSEE instrument which would incorporate the new understanding into its design. The instrument first authorized was a scanning instrument which would be suitable for examining an entire solid rocket motor case segment (about 800 square feet of area) with a resolution of 1 inch in a time of 20 minutes. This procedure was chosen for compatibility with the existing inspection, which is done with a resolution of 6 inches. The linear speed of this inspection is 75 feet/minute. The scanning instrument consisted of a six-channel linear array of OSEE sensors arranged with a single lamp and suitable for mounting on a robotic arm (Welch, *et al.*,1993; Perey, 1995). Figure 3 shows some data from tests of the demonstration unit on a test bed with a test sample made from three plates of two-inch width, the center plate of which was cleaned. The figure shows the first scan of 6 OSEE channels and the difference between the first and second scans, again for 6 channels. The responsiveness, reproducibility and dynamic range of the measurement are clearly indicated in the figure. Figure 4 shows the response values inferred from a stepped contamination sample with steps at a 1 inch spacing. Following the successful demonstration, the six-inch probe was placed in the development queue in other facilities, with the NDE Laboratory at NASA Langley Research Center assuming a supporting and consulting role.

The next demonstration project authorized was an inspection instrument which could be used for spot inspections over a 1 inch diameter area which might well be in a difficult-to-access area of the motor. It was to be an instrument which could be used practically by a single operator with access to the motor on a series of catwalks or a scaffolding used in production settings. While the intercalibration issues of the earlier instrument were avoided in the new instrument configuration, other issues were addressed associated with weight, portability, manipulability, establishing the purge, confirming measurement geometry prior to a measurement, event timing for a single measurement and operator feedback (Perey, 1997). This instrument is configured as a small base unit, a small tank of argon and an inspection "gun" (Figure 5) at the end of an umbilical. It has several modes of operation, including a single spot measurement, a continuous measurement mode, in which the position can be varied, and an automatic mode appropriate for robotic inspections. This instrument is expected to be operational as a demonstration unit late this summer.

6. OSEE application tests.

At the present level of development, OSEE has been found to be very sensitive to certain kinds of contamination. As a rule, it seems from several studies of substrate-contaminant pairs that greases and hydrocarbon films on metal substrates are good candidates for OSEE inspection. In the studies, when OSEE is sensitive to contamination, the level of sensitivity has been found to be less than 1 $\mu g/cm^2$ (the limit of our ability to control contaminant thickness on samples) or on the order of $\mu g/cm^2$. However, OSEE has also been found to be relatively insensitive or even confusing with some contaminant-substrate pairs. In view of the variability in sensitivity and lacking a complete physico-chemical model of OSEE response to contaminants, it is appropriate to perform a responsiveness study to likely contaminants in each inspection setting for which OSEE inspection is being considered. Several such studies have been done to date, and the beginning of an OSEE sensitivity library could be formed. The OSEE measurement portion of these studies is anticipated to become substantially faster, cheaper and more convenient with the completion of the instrument under development at NASA Langley Research Center.

7. Future research and development

The virtues of OSEE for surface inspection are that no mechanical contact with the surface is required, that its reading is immediate on inspection and that it can be performed in factory environments. This makes OSEE very attractive for production settings.

With even a simple low pressure mercury lamp, OSEE response comes from two widely separated spectral lines. These may be called high energy (for the 185 nm line) and low energy (for the 254 nm line) OSEE. From some experimental observations and theoretical considerations, it is reasonable to suppose that the two responses indicate different surface properties. For example, the high energy response may be more sensitive to contamination film thickness while the low energy response may be more sensitive to the work function of the substrate. Exploring the spectral response of OSEE has the potential to broaden the surface characterizations which can be addressed with OSEE inspections.

Surface science has developed a host of techniques which can describe surface films and particulate contamination, in many cases, to the level of a few atoms. Some of these techniques use the same photoelectrons that OSEE uses, but have the additional ability to describe the energy and polarization of the emitted electrons. These techniques generally require substantial care in sample preparation, and samples have to be extracted which can be placed in high vacuum chambers. It would make sense to use the power of surface science techniques to verify hypotheses about the operation of OSEE, so that a theory of its sensitivity can be developed and refined.

One surface science method, with commercially available equipment called PEEM, produces data related to OSEE, using ultraviolet light and collecting photoelectrons. This came from earlier work, such as that of Baxter and Rouze (1973), on an instrument called a photoemission electron microscope. This delelopment shows clearly that microscopic features of interest are visible with variability in photoelectron emission. Some correlation has been found with fatigue processes and the formation of slip lines, attributed to fractures of oxide layers. Most of this work uses electron imaging lenses to obtain the images of photoelectron emission variations, and so these methods must be used in a high vacuum environment, to permit undisturbed electron trajectories. To develop comparable data in the ambient pressure environment of nondestructive testing, a scanning OSEE system similar to those in the new instruments is an appropriate development goal.

8. Summary

Optically Stimulated Electron Emission (OSEE), a surface inspection technique introduced by NASA and its contractors to address immediate problems in the manufacture of the Space Shuttle, seems to have untapped potential as an inspection device for many production settings, where surfaces have just been prepared prior to forming bonds. The failure of such bonds has been shown in many cases to be due to surface contamination, and OSEE provides a rapid, non-contact method of assessing the surface. To tap the potential, application studies are needed, These studies can be greatly facilitated by a new instrument which incorporates what has been learned in recent studies of OSEE operation.

References:

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Yost, W. T., C. S. Welch, E. J. Joe and B. B. Hefner, *Quality Monitor and Monitoring Technique Employing Optically Stimulatee Electron Emission*, U. S. Patent No. 5,393,980, February 28, 1995.

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Welch, C., W. Yost, S. Gasser, E. Joe, D. Perey, E. Scales, J. Bly, T. Goodman, W. Hefner, X. Pascual, F. Stone, C. Kennedy and B. Batten, "OSEE Instrument: Design and Construction," Technical Interchange Meeting, Huntsville, AL, (1993, unpublished).

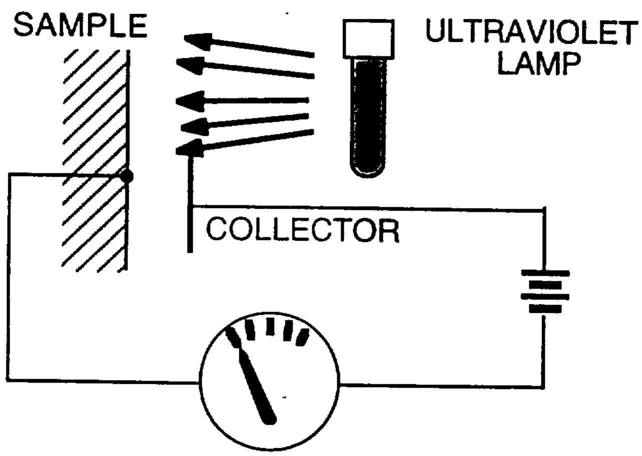

Figure 1. Schematic circuit of an OSEE instrument (after Gause, 1989). Shown is a direct current circuit with a battery, a means of measuring current and a surface illuminated with an ultraviolet light. The circiut is completed by the photoelectrons which cross the gap to be collected at the positively charged anode.

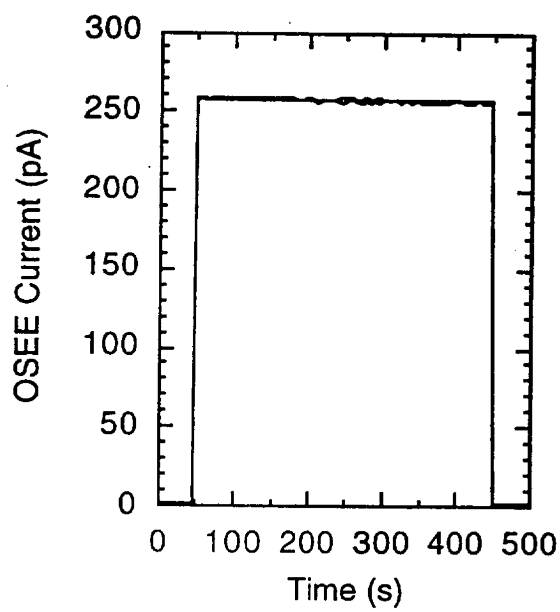

Figure 2. Two superimposed curves of OSEE current vs time. These data, from a copper sample in an argon atmosphere, show the degree of reproducibility which can be obtained with OSEE in favorable circumstances.

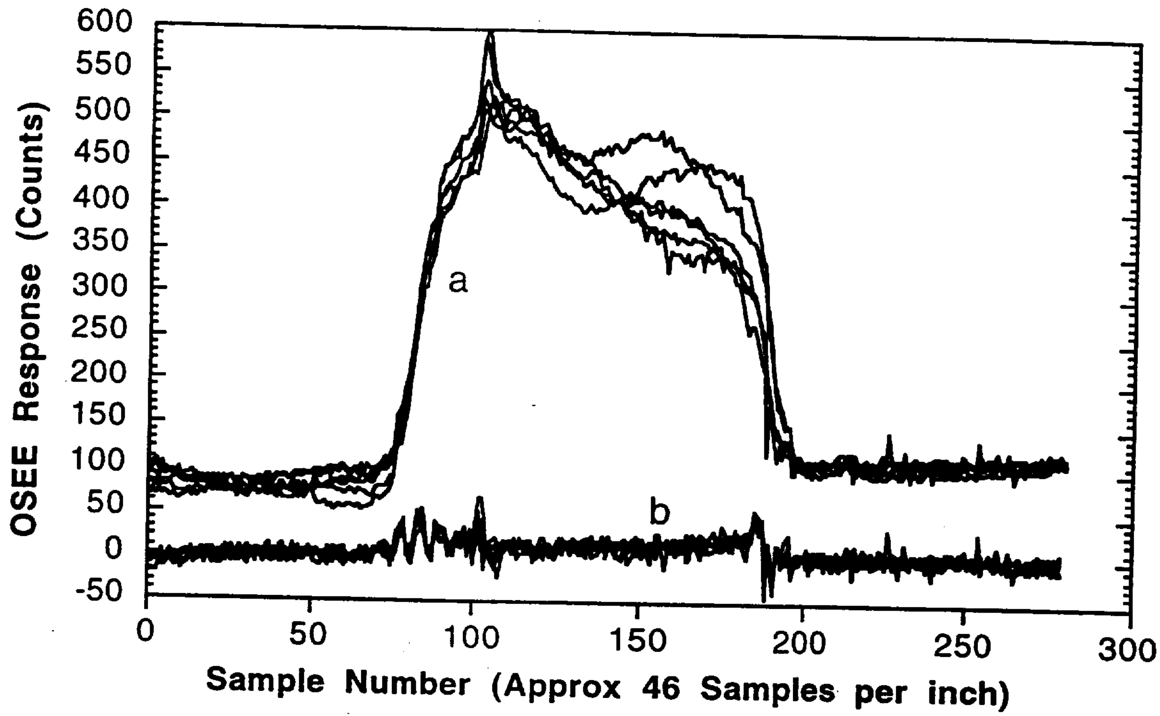

Figure 3. Results of two successive scans of the 6-channel OSEE instrument over a test object consisting of three plates, the central one being clean and the two outer plates being dirty. The results in a are the data for all 6 parallel channels from one of the scans, while those in b are the differences between the two scans. The largest differences, in the high gradient region of the data, are attributed to differences in scanner position rather than differences in OSEE readings.

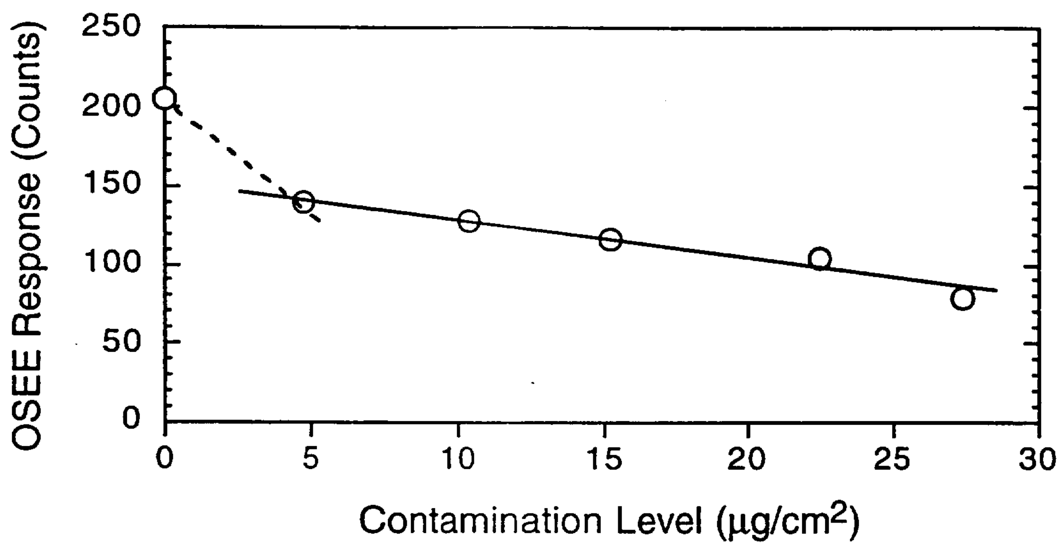

Figure 4. Averaged OSEE data over a stepped sample of varying contamination amounts. The dotted line between the clean area and the nominal 5 µg/cm^2 indicates an increase in sensitivity in that region.

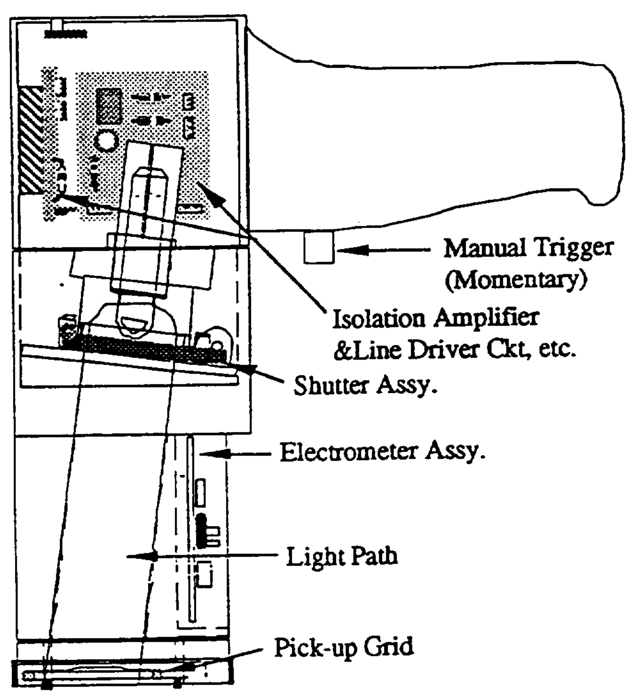

Figure 5. Cross-section of the probe head in the first prototype of the hand-held OSEE instrument. The light path is about 1 inch in diameter. (after Perey, 1997)

Ultrasonic Nondestructive Characterization of Adhesive Bonds

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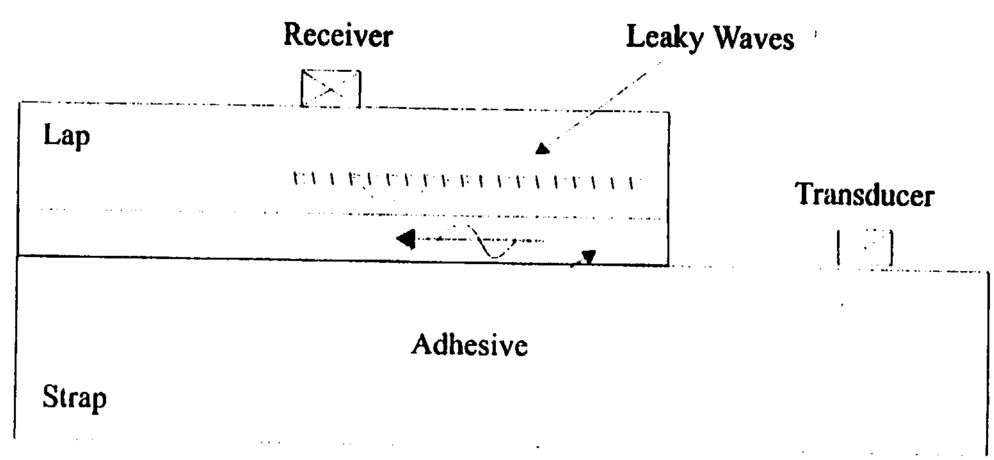
Atlanta, GA 30332

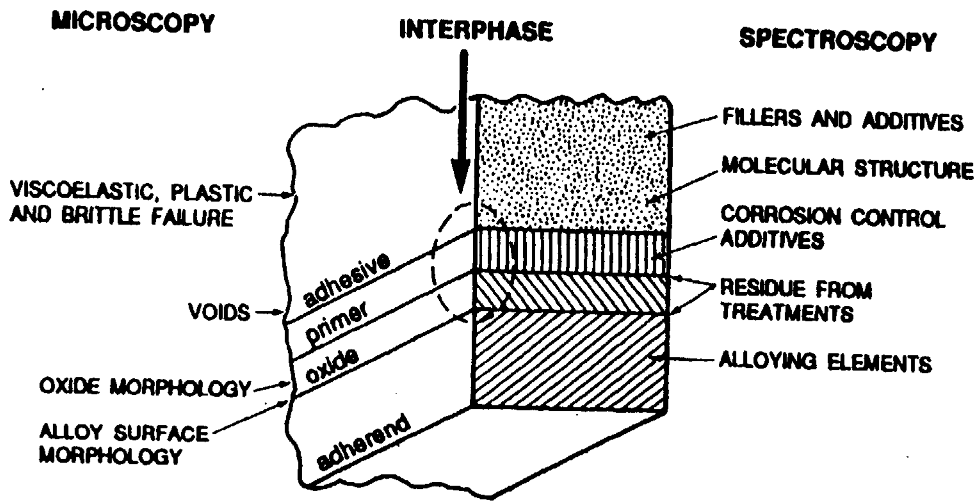
Objectives

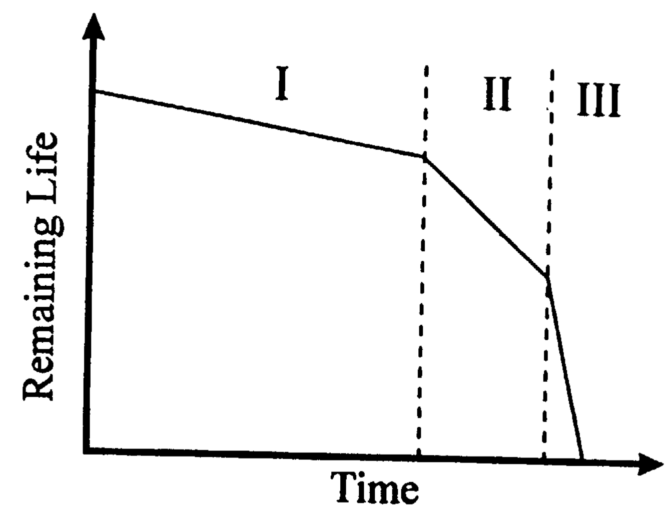
This project is concerned with the qualification of reliability and integrity of metal/polymer bond joints. The objectives are

* To establish the correlation between the microstructural changes and ultrasound propagation characteristics.
* To develop ultrasonic nondestructive methods to measure the microstructural changes caused by the degradation of bond strength.
* To predict remaining bond strength from ultrasonic measurement based on the fundamental structure-property-performance relationships of the constituents and their interfaces.



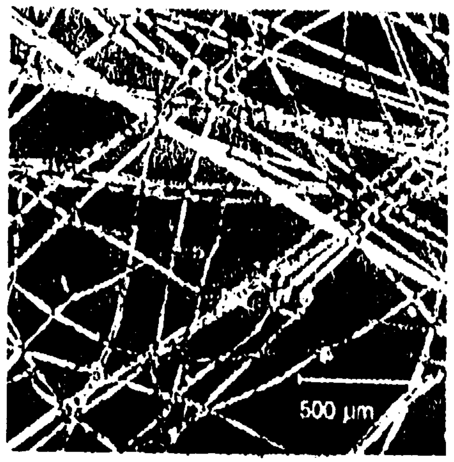
Degradation, Damage and Failure





	Degradation	Damage Accumulation	Failure
Adhesive	*crosslink density* *moisture absorption* *plasticization*	*creep and fatigue* *microvoids* *microcracks*	*fracture* *crazing*
Interphase	*corrosion* *oxidation*	*microvoids* *microcracks*	*debonding* *delamination*
Adherend		*deformation*	*fatigue and fracture*

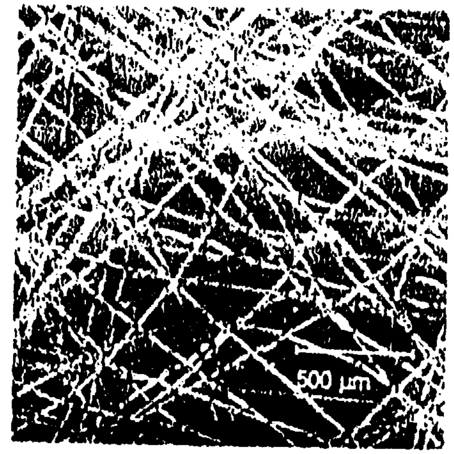
Scrim Cloths



FM-73M

A toughened epoxy adhesive containing a non-woven polyester scrim cloth (CYTEC Engineered Materials, Inc.)

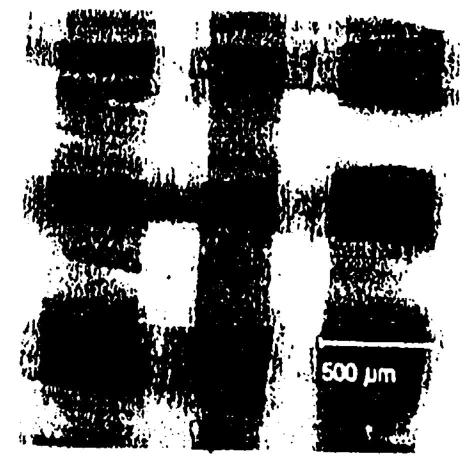
Service T < 82C.



AF-191M

A modified epoxy adhesive containing a non-woven nylon scrim cloth (3M Corporation.)

Service T < 177C

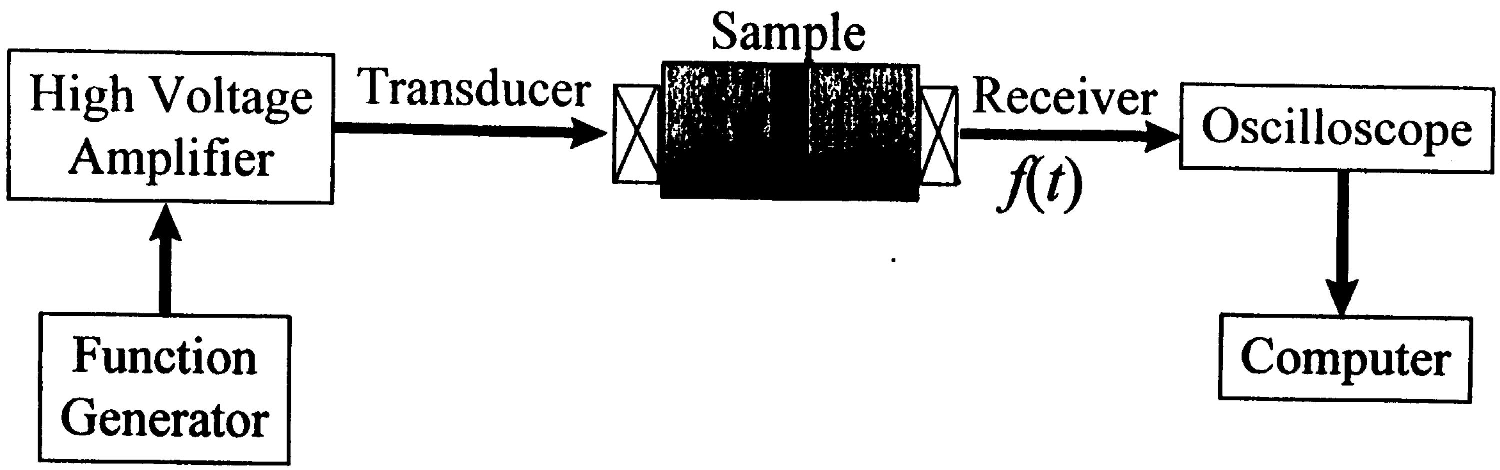


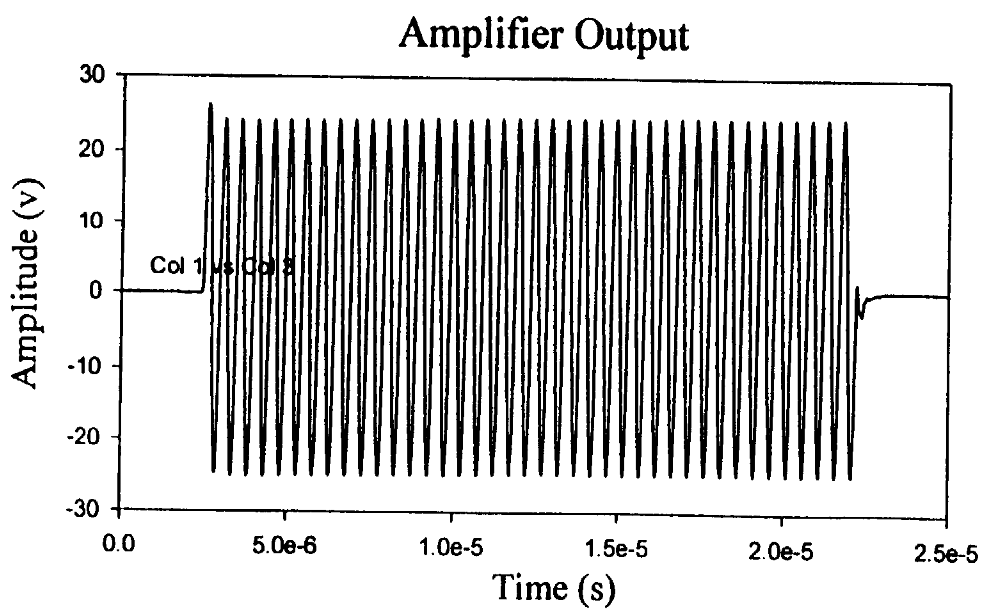
FMx5

A semi-crystalline amorphous polyimide blend of PETI-5 and other thermoplastic resin containing a woven glass scrim cloth (CYTEC Engineered Materials, Inc.)

Service T < 177C

Through Transmission



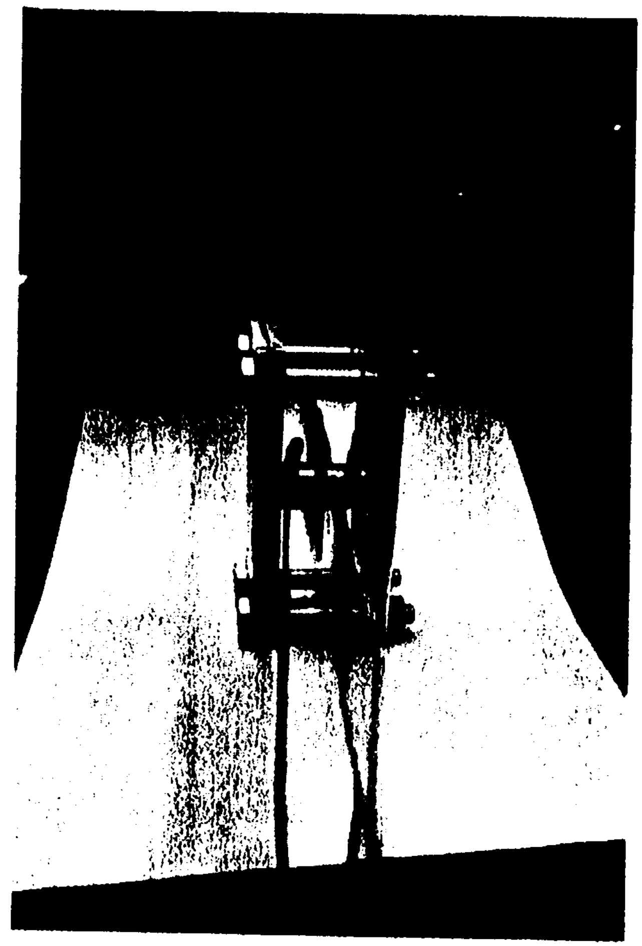


Generation

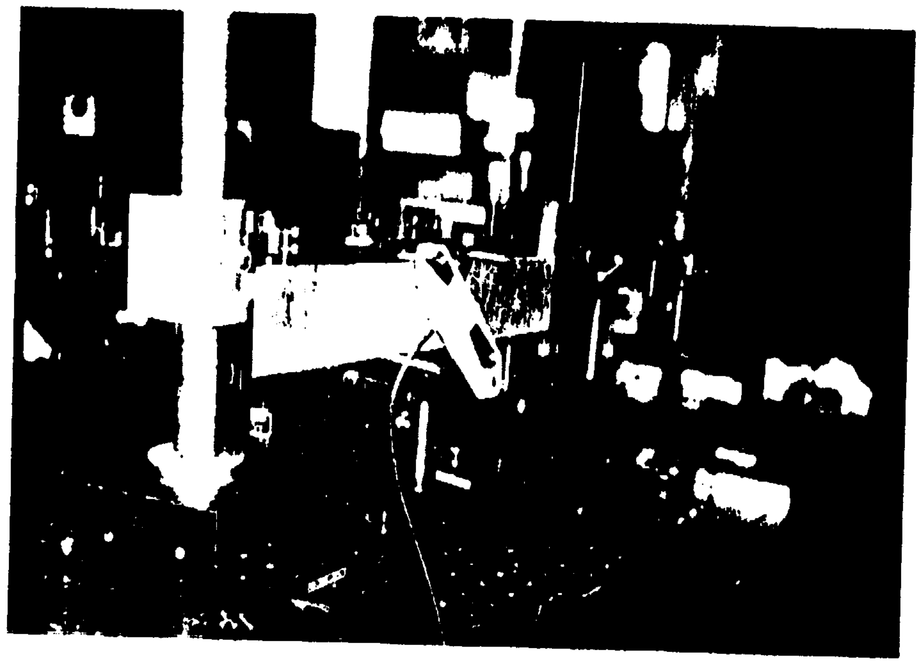
* PZT (2 and 5 MHz)
* Single Crystals (quartz, lithium niobium)

Detection

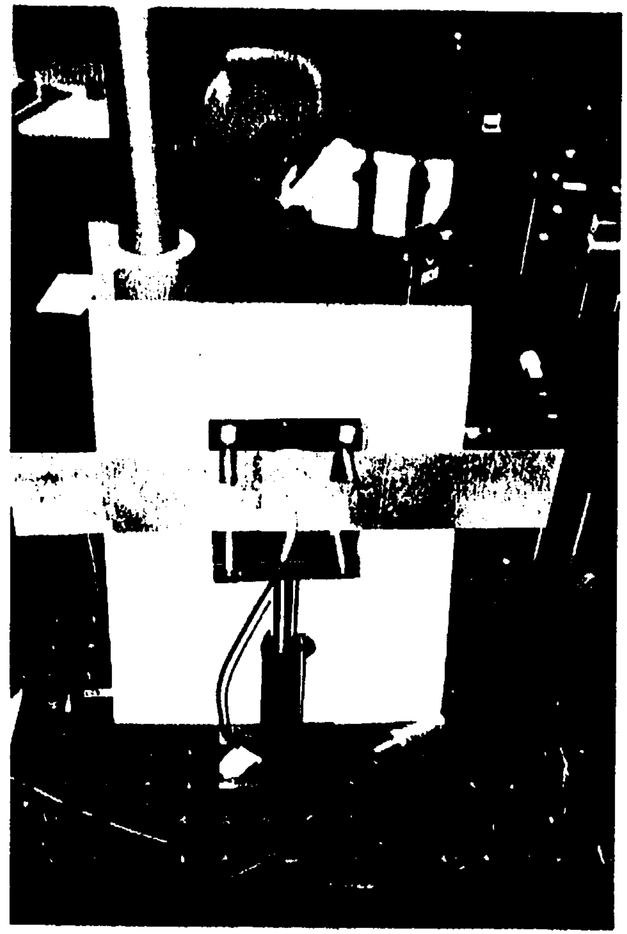
* PZT (2 and 10 MHz)
* Laser Interferometer



PZT/PZT



PZT-Laser



Single Crystal - PZT

Equipment

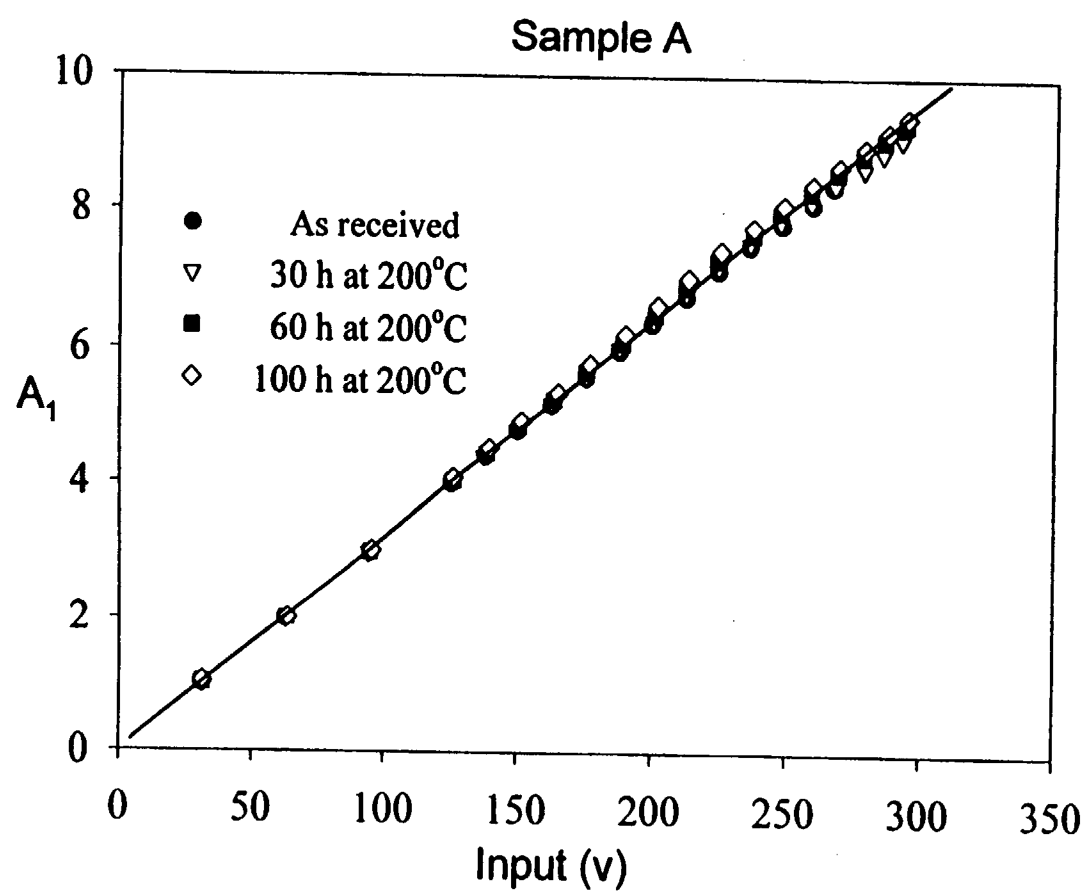
Transducer: 2 MHz, narrow band PZT
5 MHz, narrow band PZT
single crystal quartz
single crystal lithium niobium

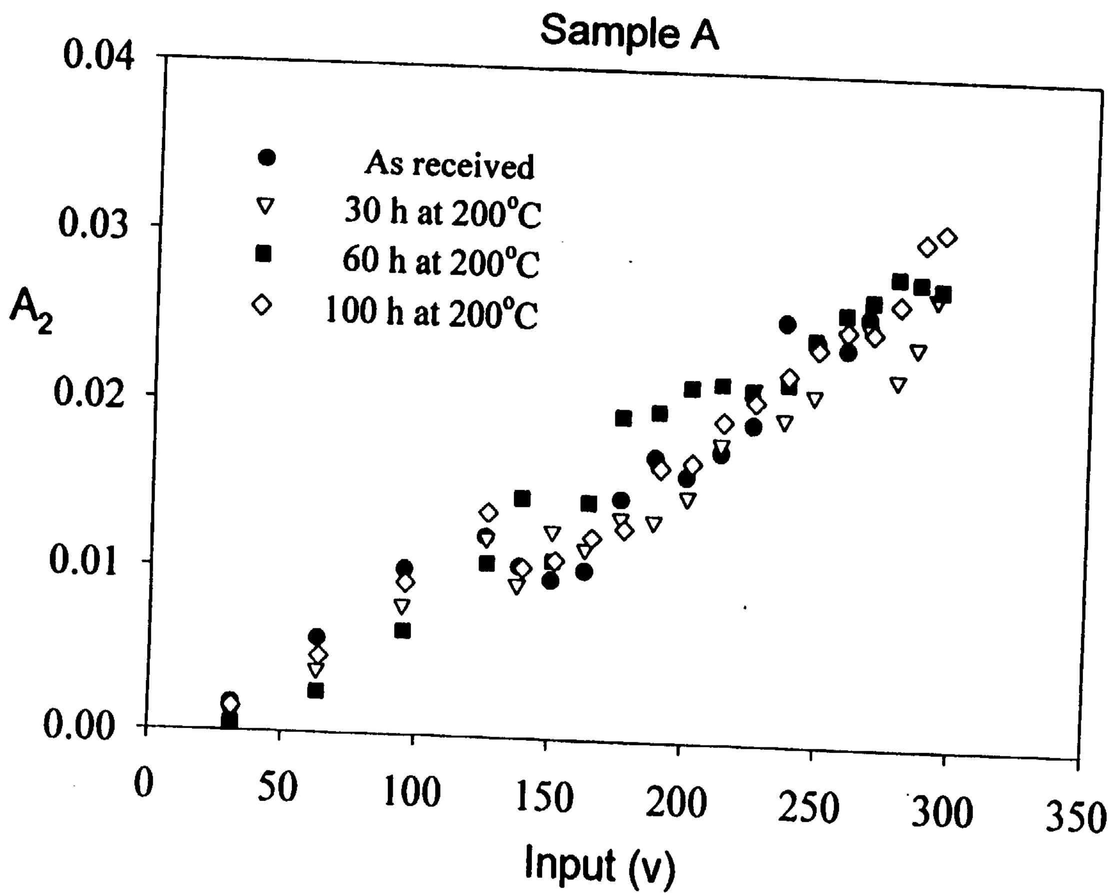
Receiver: 2 MHz, broad band PZT
10 MHz, narrow band PZT
Laser Interferometer

Amplifier: ENT, DC ~ 10 MHz, 50dB

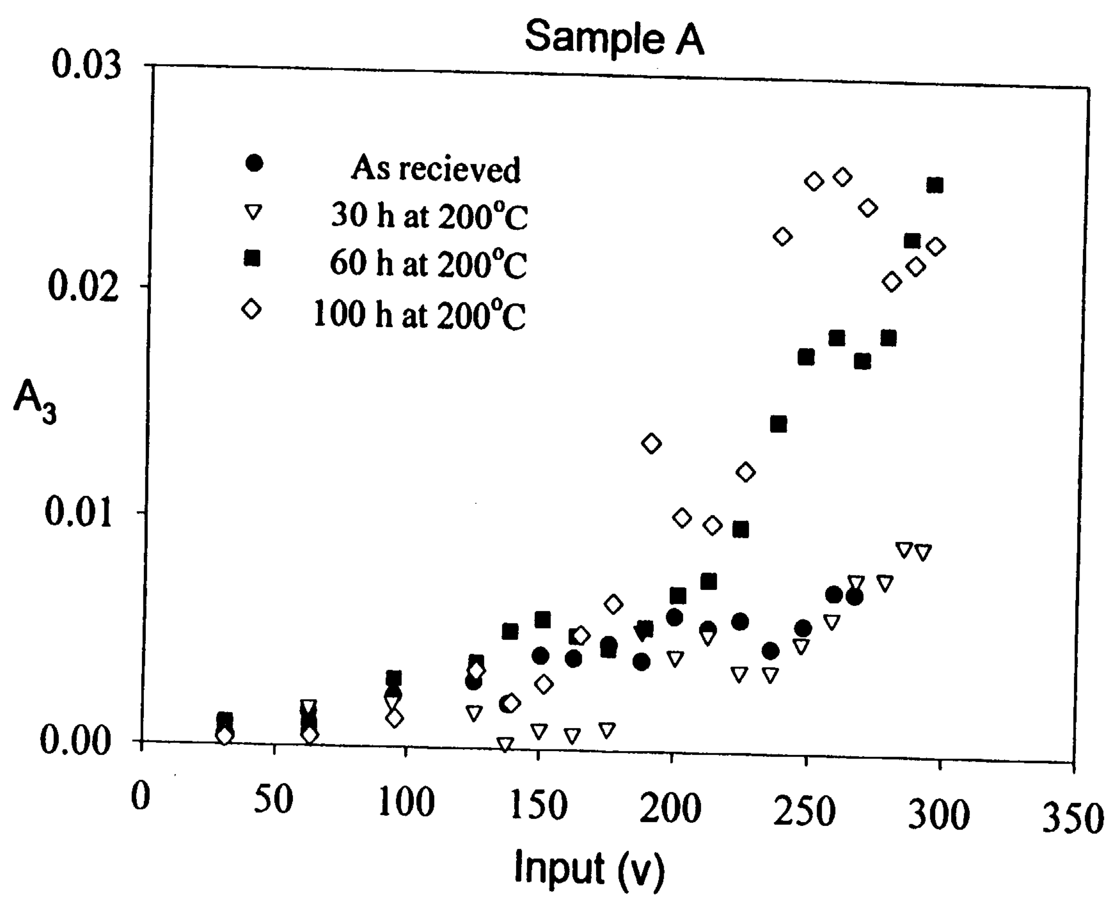
Function Generator: Wavetek, 50 MHz

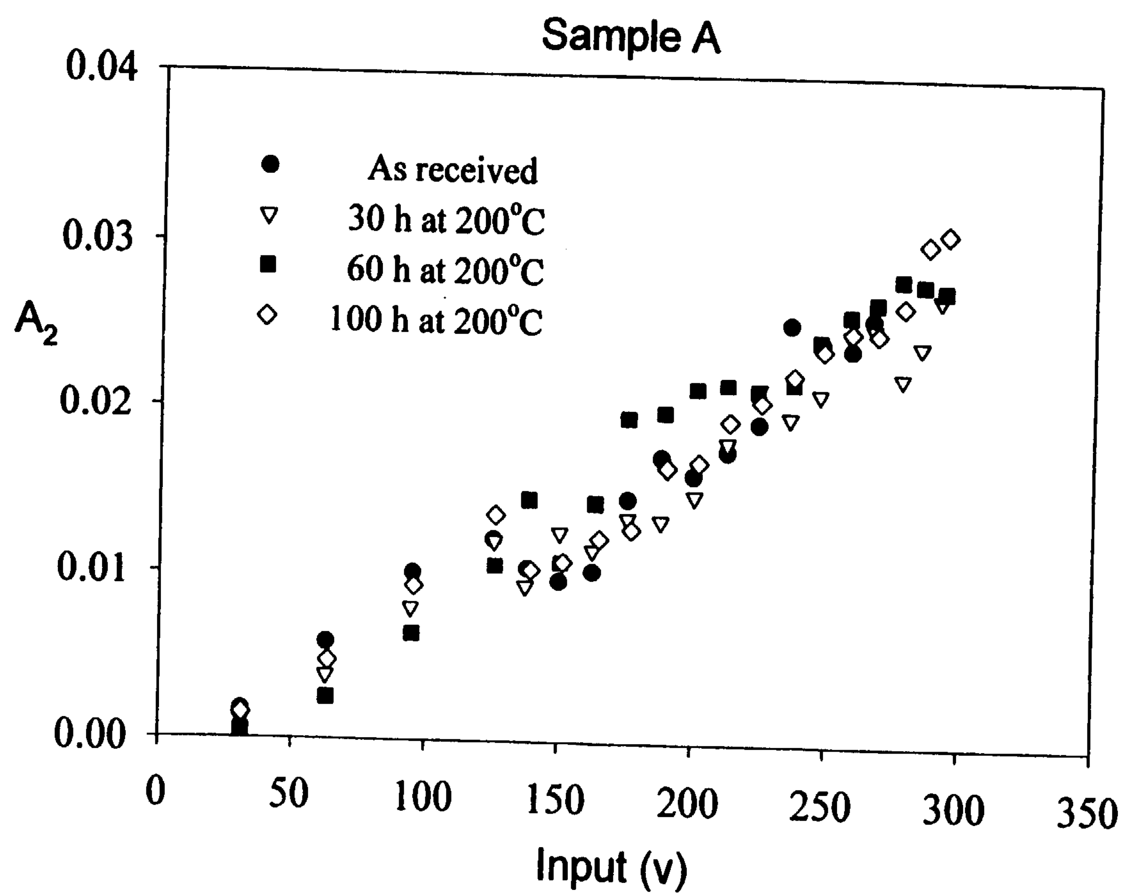
Oscilloscope: Techtronix, 150 MHz

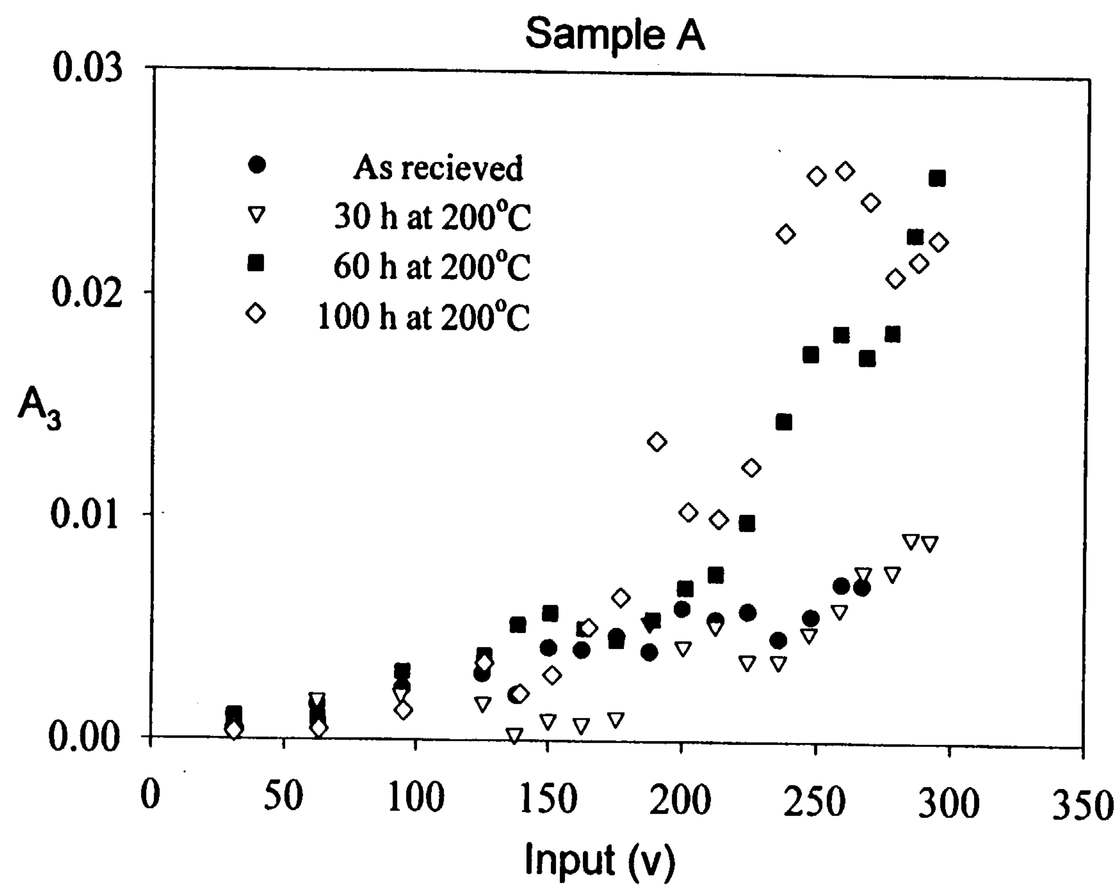

Sample A
10
8
6
4
2
0
A1
As received
30 h at 200°C
60 h at 200°C
100 h at 200°C
0
50
100
150
200
250
300
350
Input (v)



Sample A
As received
30 h at 200°C
60 h at 200°C
100 h at 200°C
A2
0.04
0.03
0.02
0.01
0.00
0
50
100
150
200
250
300
350
Input (v)

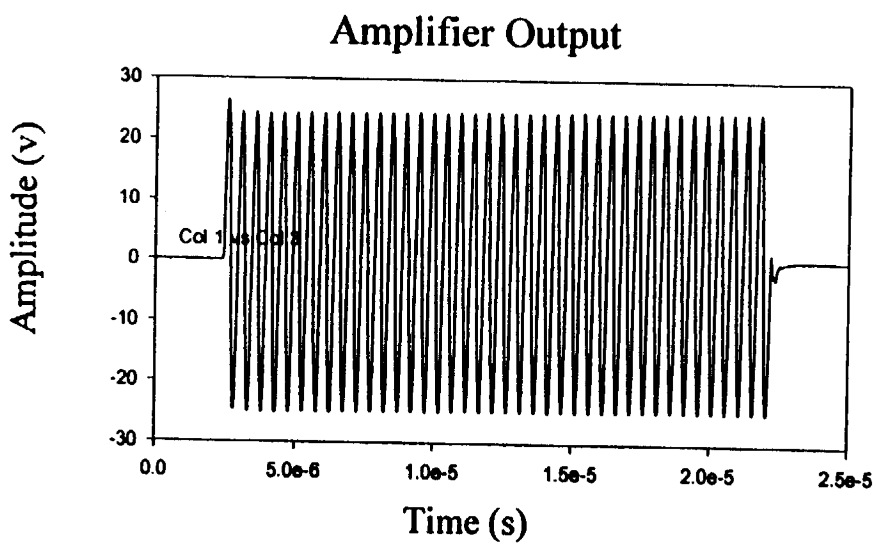

Sample A
0.03
0.02
0.01
0.00
A_3
0
50
100
150
200
250
300
350
Input (v)
As recieved
30 h at 200°C
60 h at 200°C
100 h at 200°C

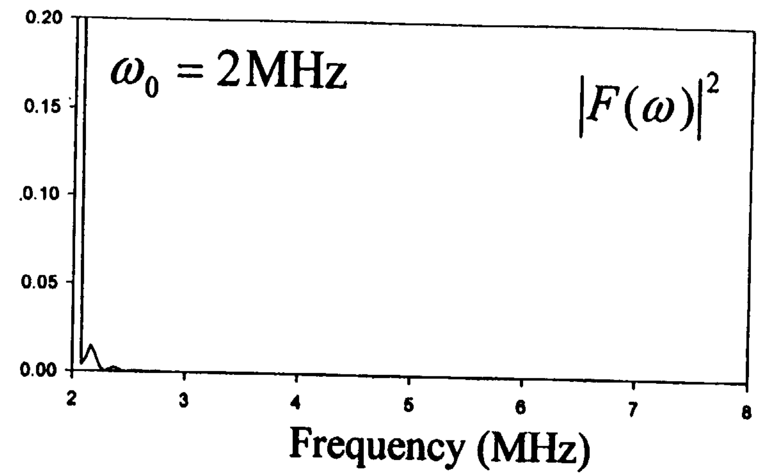

Sample A
0.04
0.03
0.02
0.01
0.00
A2
As received
30 h at 200°C
60 h at 200°C
100 h at 200°C
0
50
100
150
200
250
300
350
Input (v)

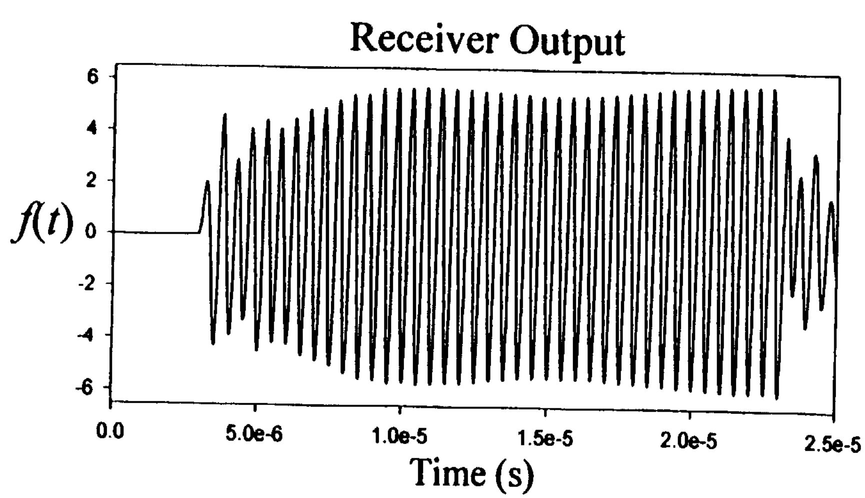

Sample A
0.03
0.02
0.01
0.00
A_3
0
50
100
150
200
250
300
350
Input (v)
As recieved
30 h at 200°C
60 h at 200°C
100 h at 200°C

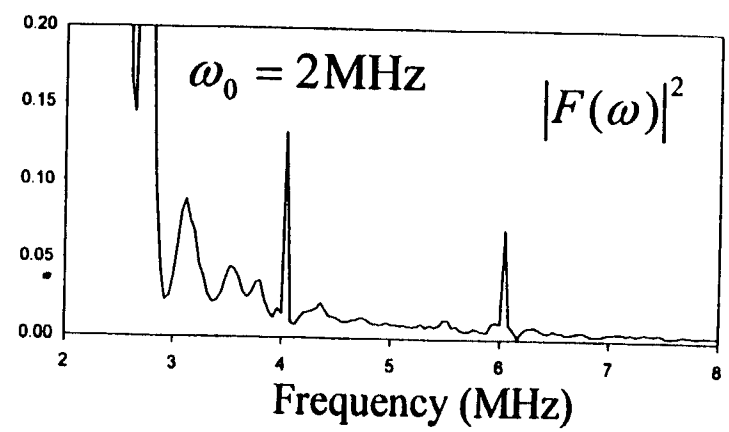
Fourier Transforms

$$F(\omega) = \int_0^\infty f(t)\exp(i\omega t)dt \qquad A_n = |F(n\omega_0)| \qquad n = 1,2,3\cdots$$

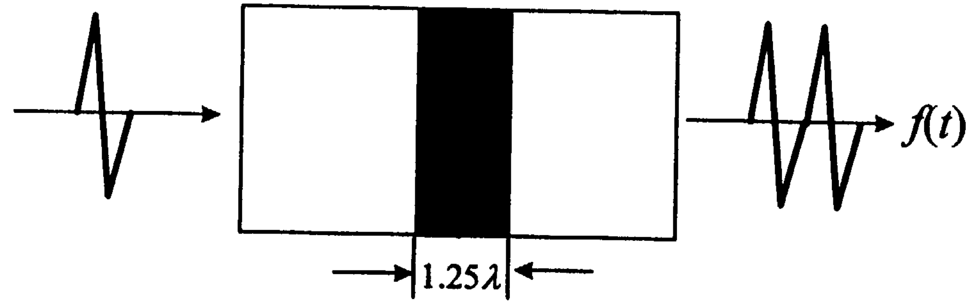




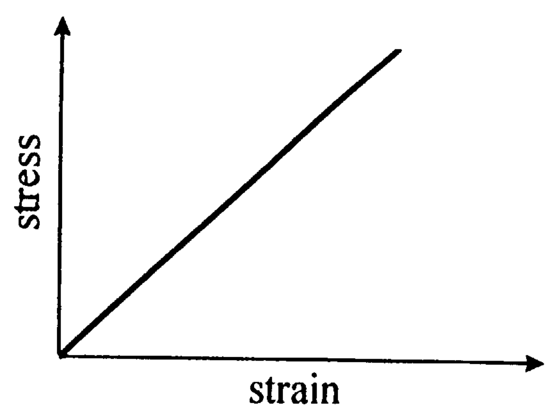




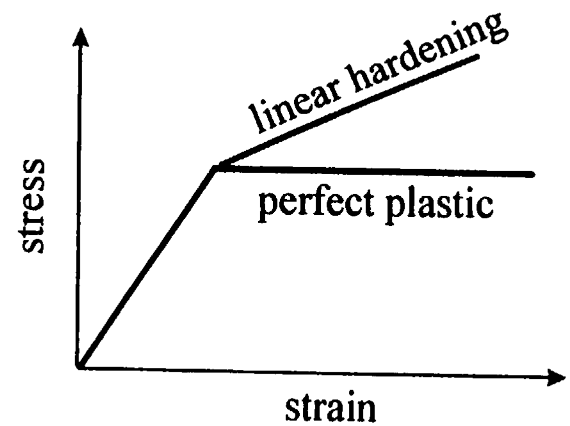
Finite Element Analysis

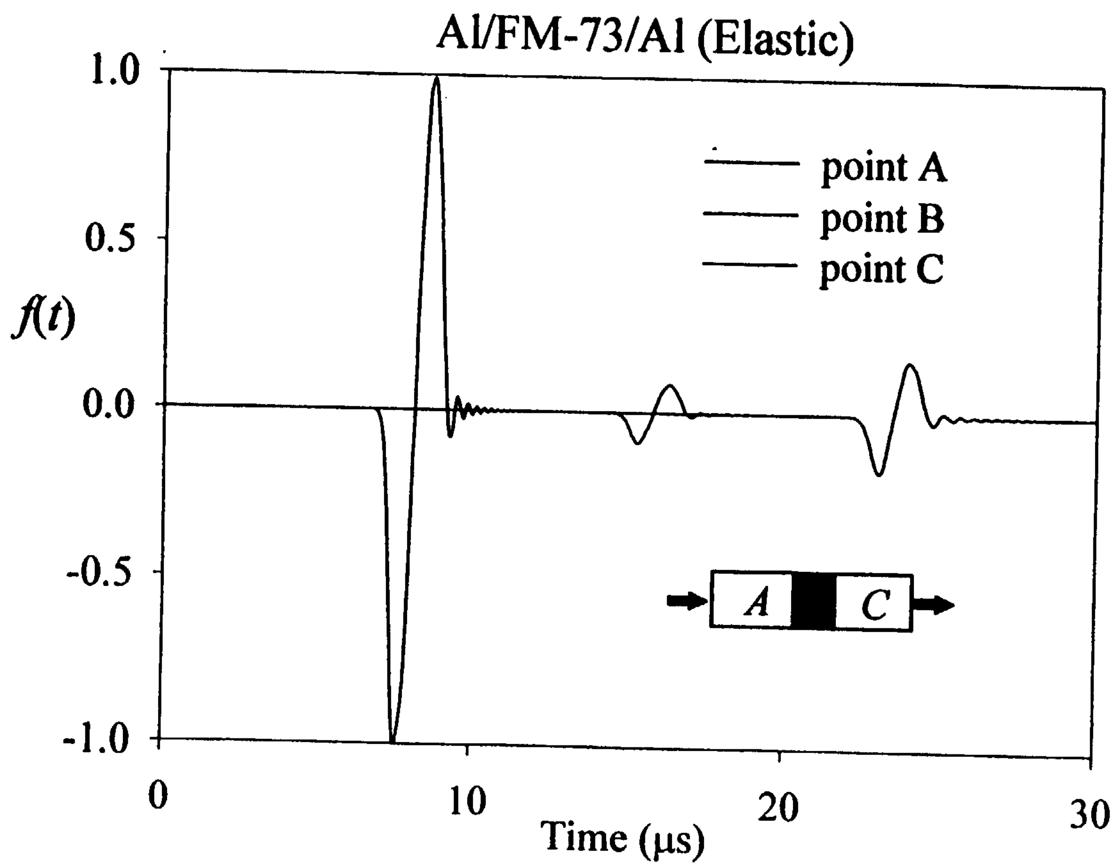


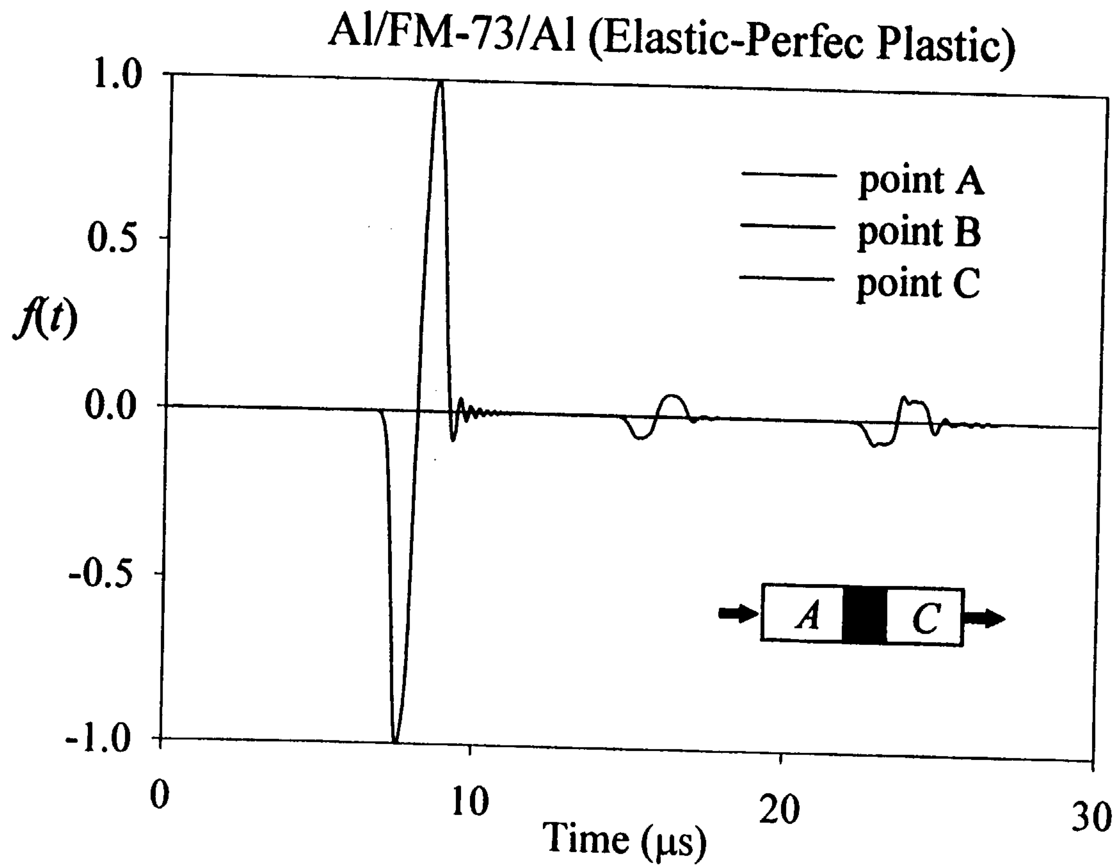
Al

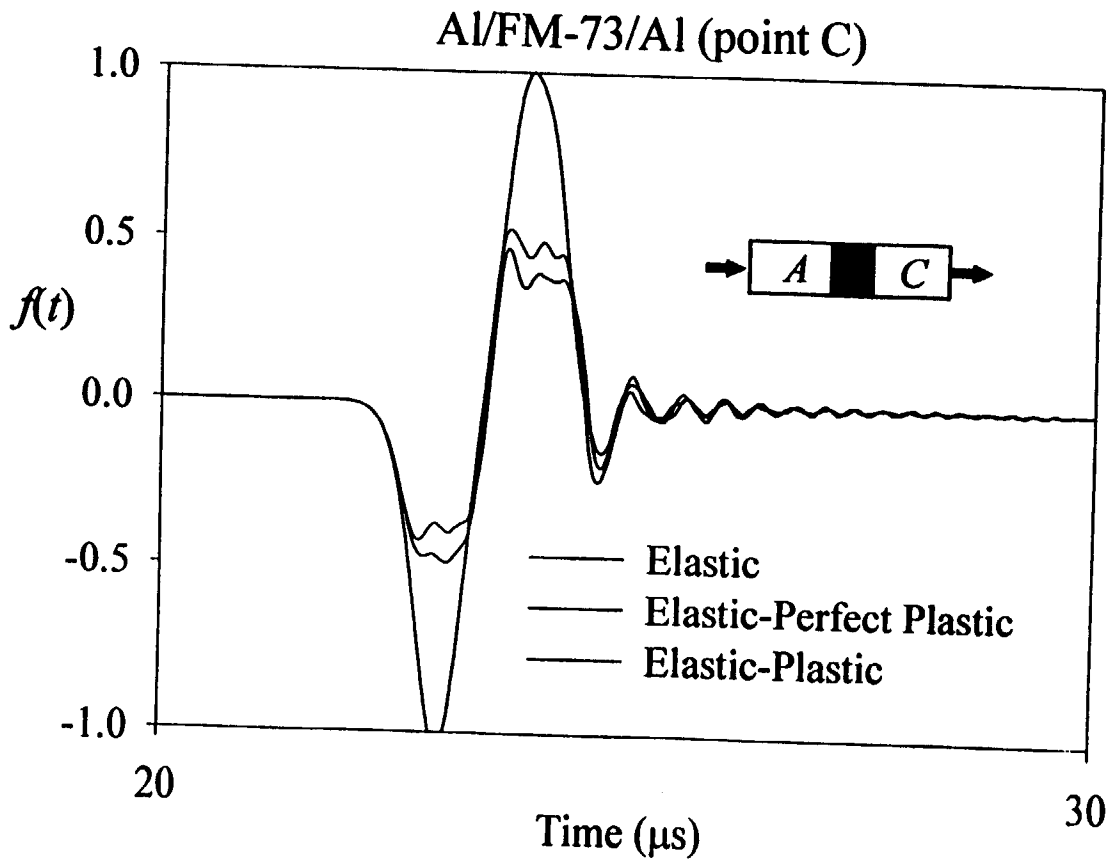


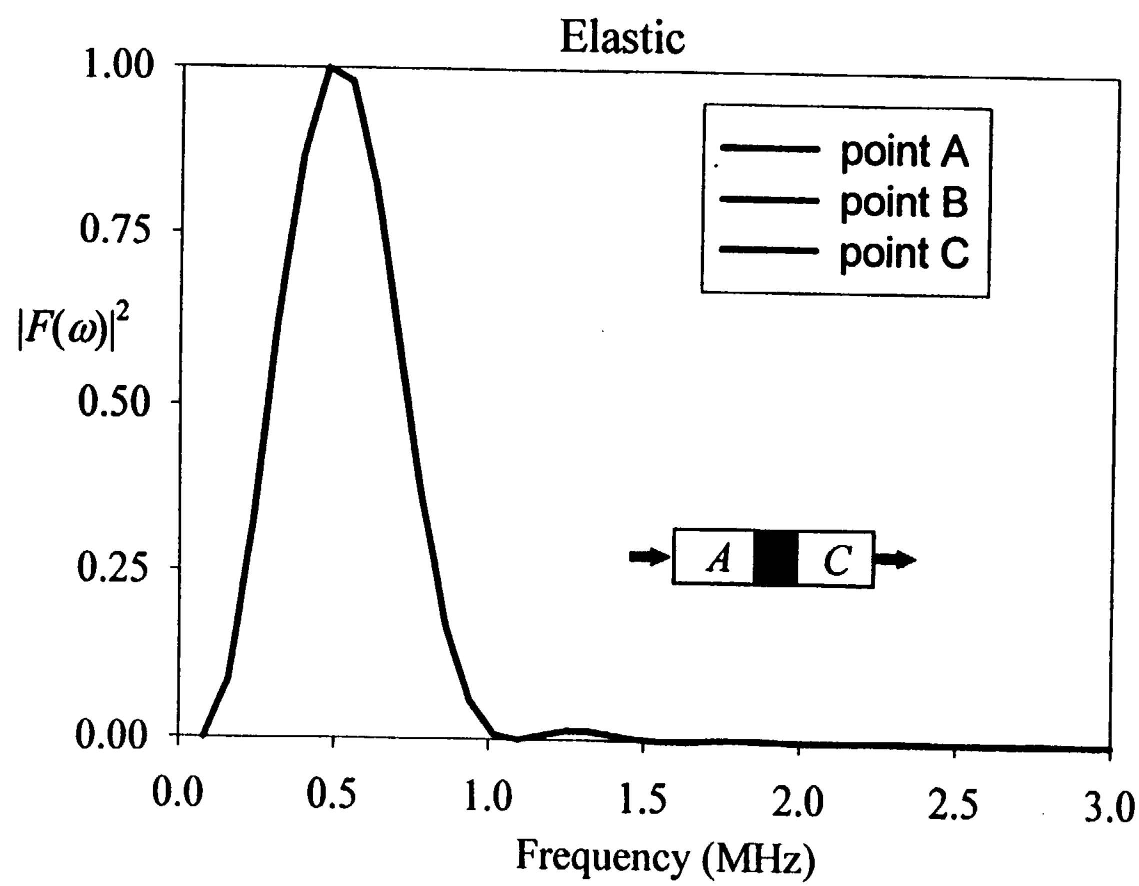
FM-73

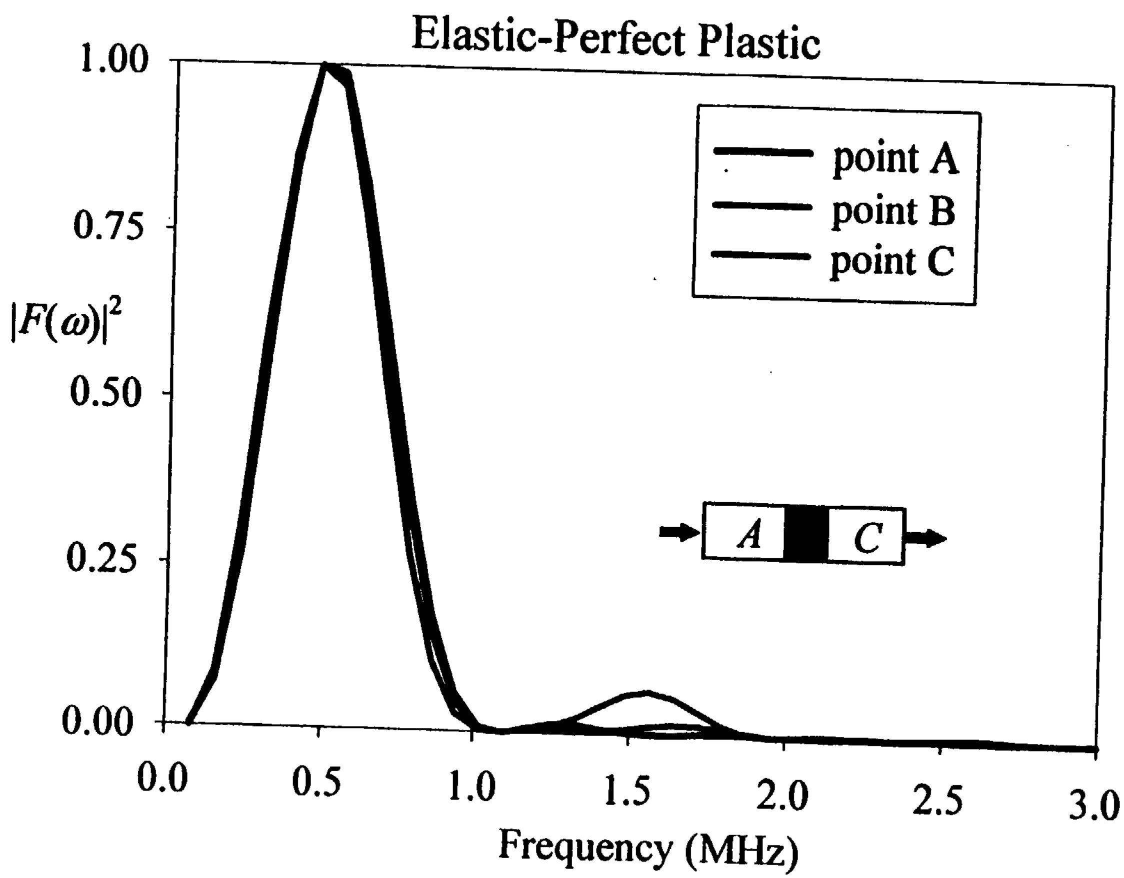



Al/FM-73/Al (Elastic)
1.0
0.5
0.0
-0.5
-1.0
f(t)
point A
point B
point C
A
C
0
10
20
30
Time (μs)


Al/FM-73/Al (Elastic-Perfec Plastic)
1.0
0.5
0.0
-0.5
-1.0
f(t)
point A
point B
point C
A
C
0
10
20
30
Time (μs)

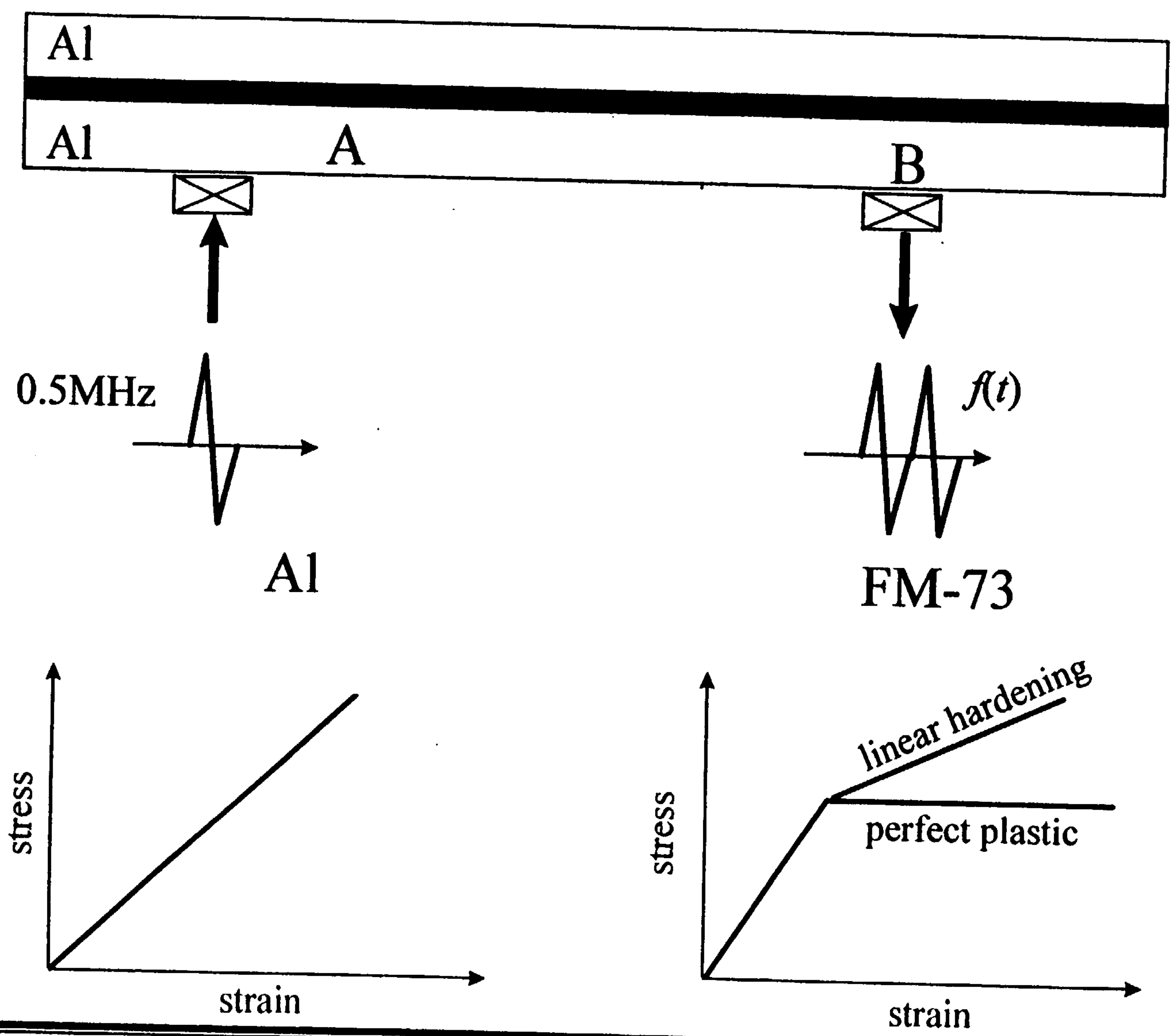

Al/FM-73/Al (point C)
1.0
0.5
0.0
-0.5
-1.0
$f(t)$
20
30
Time (μs)
A
C
Elastic
Elastic-Perfect Plastic
Elastic-Plastic


Elastic
1.00
0.75
$|F(\omega)|^2$
0.50
0.25
0.00
0.0
0.5
1.0
1.5
2.0
2.5
3.0
Frequency (MHz)
point A
point B
point C
A
C

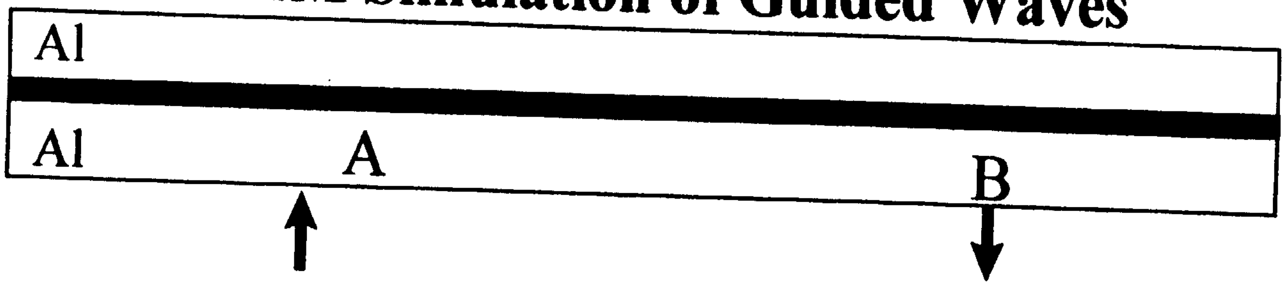

Elastic-Perfect Plastic
1.00
0.75
0.50
0.25
0.00
$|F(\omega)|^2$
0.0
0.5
1.0
1.5
2.0
2.5
3.0
Frequency (MHz)
point A
point B
point C
A
C

FEM Analysis of Guided Waves

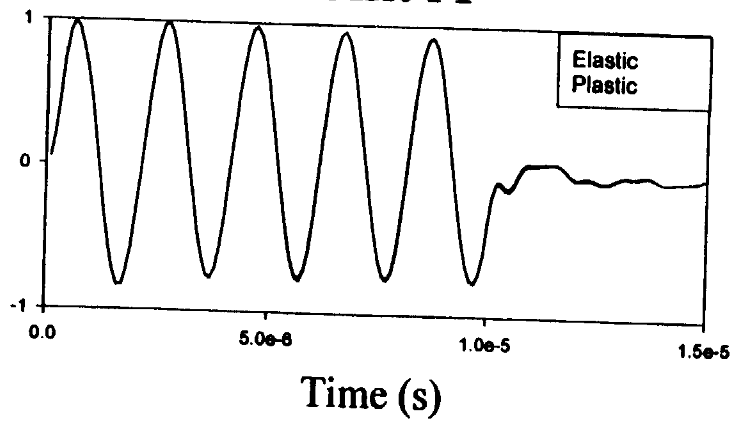
(Al/ FM-73/Al)



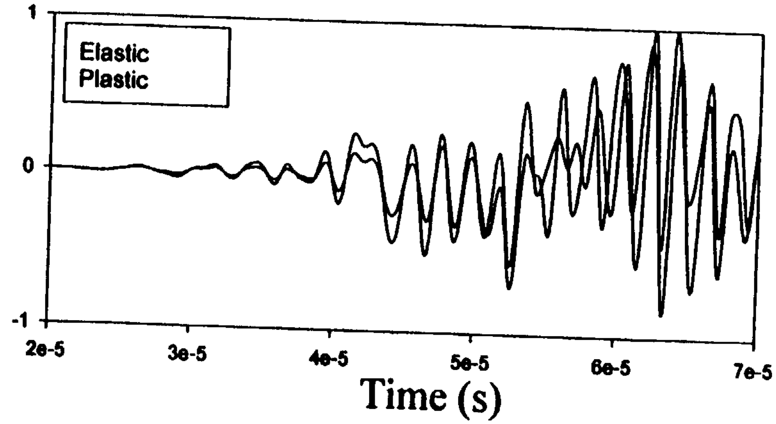
FEM Simulation of Guided Waves



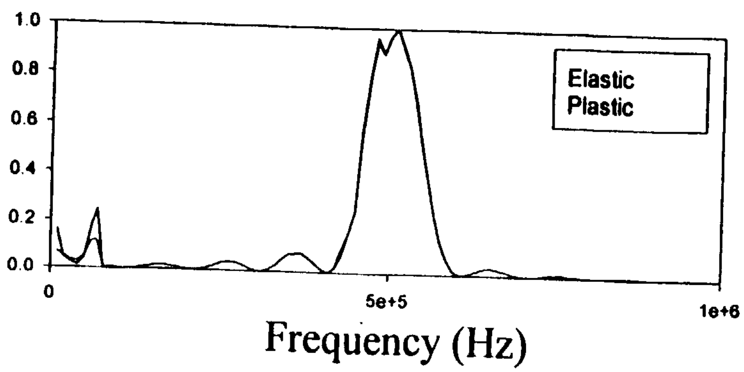
Point A



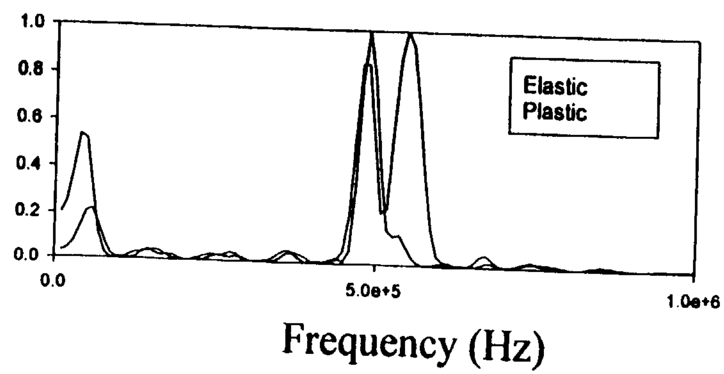
Point B

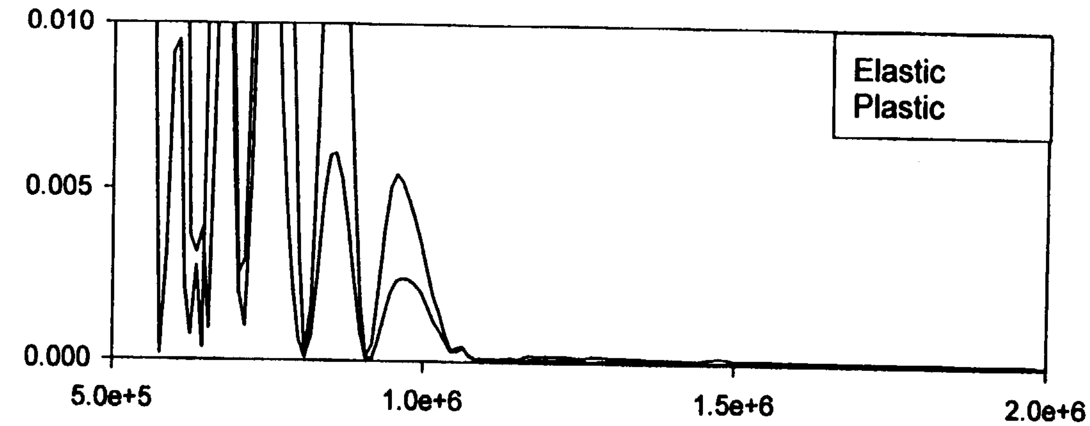


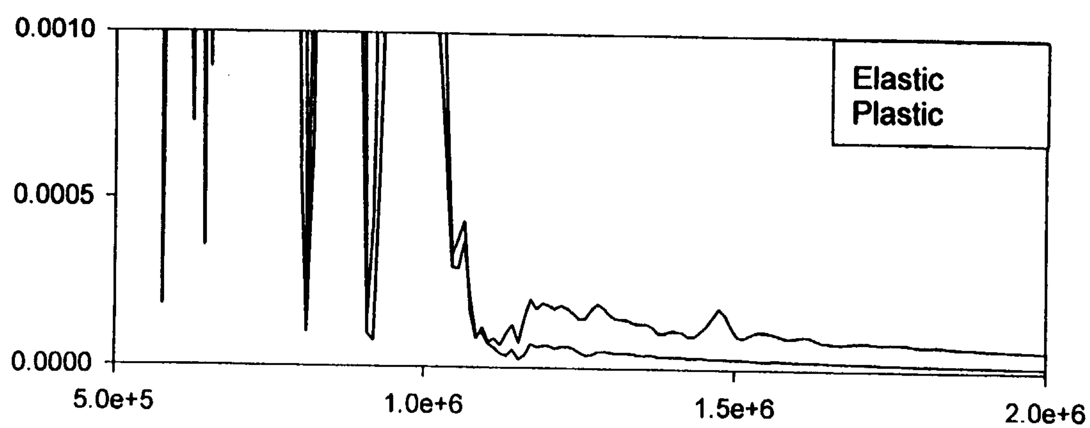
$f(t)$



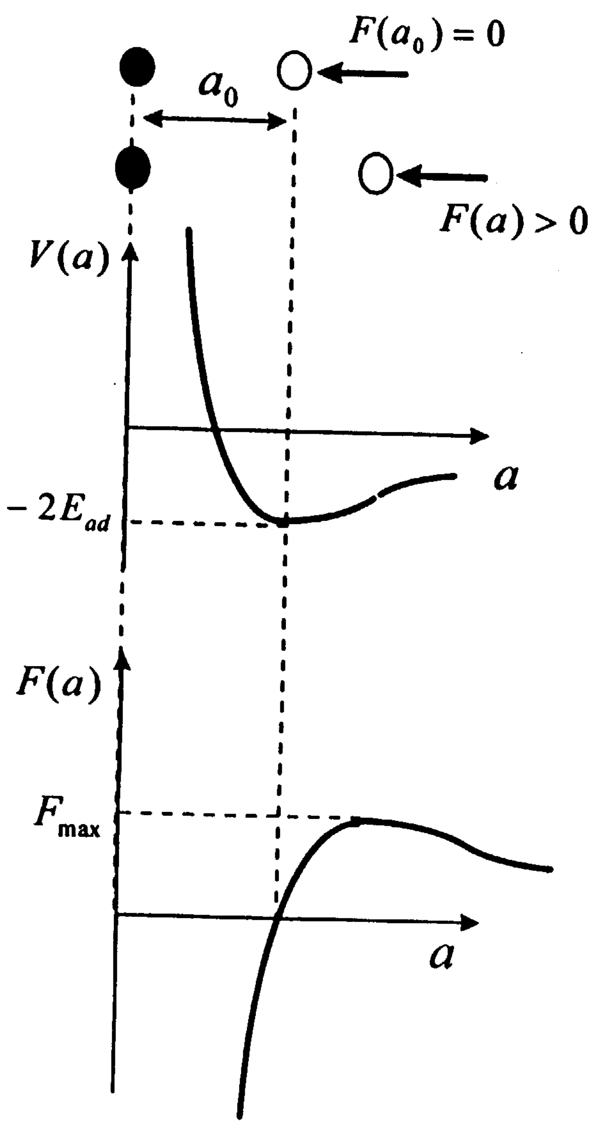
$|F(\omega)|^2$




0.010
0.005
0.000
5.0e+5
1.0e+6
1.5e+6
2.0e+6
Elastic
Plastic


0.0010
0.0005
0.0000
5.0e+5
1.0e+6
1.5e+6
2.0e+6
Elastic
Plastic

Interfacial Binding Force



Binding Energy

$$V(a) = \frac{2E_{ad}}{l_{sc}} \exp(-\frac{a}{l_{sc}})$$

Binding Force

$$\vec{F}(a) = -2E_{ad} \frac{\vec{a}}{l_{sc}^2} \exp(\frac{-|\vec{a}|}{l_{sc}})$$

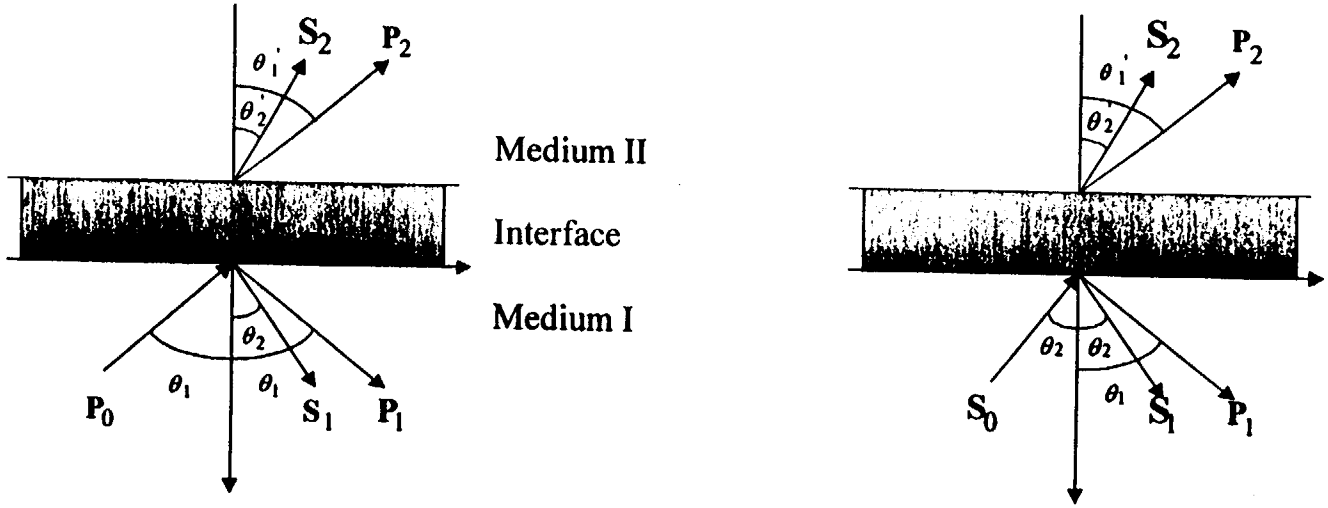
$\vec{a} = (a_x, a_y)$

$a_x = u_0 \sin\theta_i \sin\omega_0 t$

$a_y = u_0 \cos\theta_i \sin\omega_0 t + a_i$

E_{ad} = adhesive interaction energy

l_{sc} = Thomas-Fermi screening length



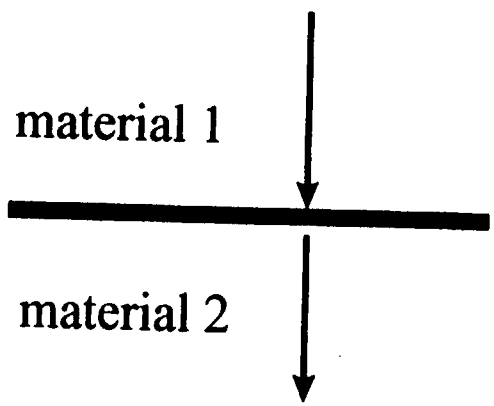
Incident P-wave　　　　Incident S-wave

$$F_x(a_x, a_y) = C_0 + C_1 \sin \omega_0 t + C_2 \cos 2\omega_0 t + C_3 \sin 3\omega_0 t + C_4 \cos 4\omega_0 t + \cdots$$

$$F_y(a_x, a_y) = D_0 + D_1 \sin \omega_0 t + D_2 \cos 2\omega_0 t + D_3 \sin 3\omega_0 t + D_4 \cos 4\omega_0 t + \cdots$$

Generation of Higher Harmonics

$$u^{in} = u_0 \sin \omega_1 t$$



$$u^t = KF(a)$$

where

$$a = a_0 + u_0 \sin \omega_1 t$$

Fourier Expansion of the Transmitted Wave

$$u^t = K\sum_{n=0}^{\infty} \{A_n \cos(n\omega_1 t) + B_n \sin(n\omega_1 t)\}$$

where A_n and B_n are functions of u_0

Thus, the binding force can be evaluated

$$F(a_0 + u_0) = \frac{u^t}{K}$$

$$= \sum_{n=0}^{\infty} \{A_n \cos(\frac{n\pi}{2}) + B_n \sin(\frac{n\pi}{2})\}$$

where t is taken as

$$t = \frac{(4j+1)\pi}{2\omega_1}, \qquad j = 0,1 \cdots n$$

Summary

Material Systems: FM-73, AF-191, FMx5 and Al/FM73/Al, Al/FM300/Al.

Chemical & Physical Analyses: FTIR, DSC, TGA

- *No significant changes were observed after various aging conditions.*

Ultrasonic Tests: Through Transmission

- *Aging increases the magnitude of higher order harmonics.*
- *Magnitude of the 3rd harmonic correlates with aging time very well.*

Finite Element Analysis: Transmission Through an Elastic-Plastic Layer

- *Material nonlinearity generates higher order harmonics.*
- *Elastic-plastic material behavior generates significant 3rd harmonic.*

Analytical Modeling: Nonlinear Spring Layer

- Inclined incidence
- Guided waves

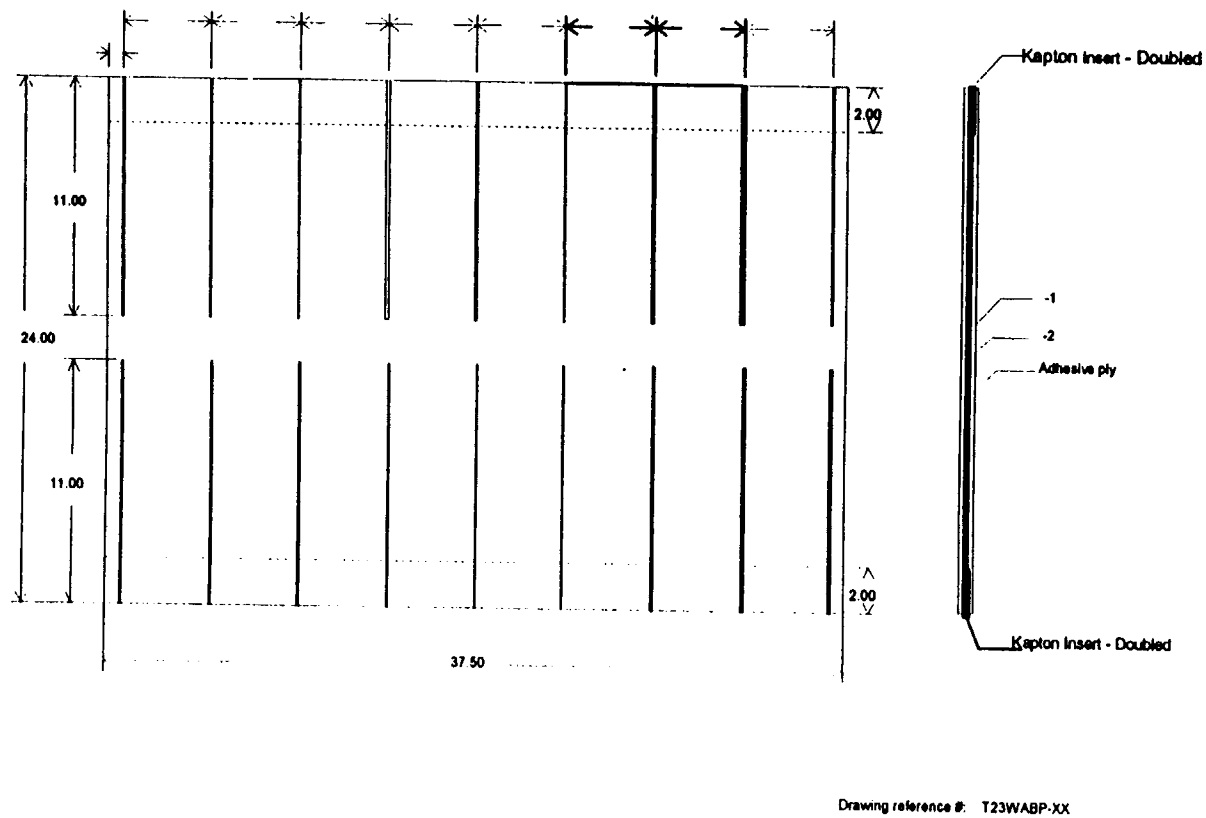
Key Issues

* Inclined incidence
* Guided waves
* Polymer/metal interfaces
* Curing and aging
* Destructive determination of interface parameters
* High power transducers (?)

Materials

Adherends: Ti-6AL4V(.10" thick) with a Sol-Gel surface preparation

Adhesive: polyimide LaRC-PETI-5 film (FMx5)



AN ULTRASONIC TECHNIQUE TO DETERMINE THE RESIDUAL STRENGTH OF ADHESIVE BONDS

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EVANSTON, IL 60208-3020

GENERAL OBJECTIVE

To develop an ultrasonic nondestructive technique to assess the adhesive bond strength of adhesive layers by analyzing the nonlinear behavior that accompanies adhesive deterioration. The work on this project is both analytical and experimental in nature.

The adhesive bond behavior can be represented by a relation between tractions, τ, and gross displacements, Δ, across the adhesive layer. Figure 1 shows four typical τ-Δ curves with their associated failure points. Figure 1a represents a brittle bond with a linear relation between τ and Δ. When τ reaches a critical value τ_{cr} the bond breaks in a brittle fashion. Deterioration of the bond gives rise to a lower value of τ_{cr}. Figure 1b shows a bond with nonlinear elastic behavior typical of rubbery adhesives. The failure point is reached for $d\tau/d\Delta=0$. Deterioration of this bond may be described by the curves shown in Fig. 1c or Fig. 1d. Note that in Fig. 1c the slope remains the same at $\tau=0$, while in Fig. 1d this slope changes. For the case of 1d the slope at $\tau=0$, which can be determined by ultrasonic methods, can be correlated with residual strength. This has been done by many investigators. Here we will address the more difficult case represented by Fig. 1c.

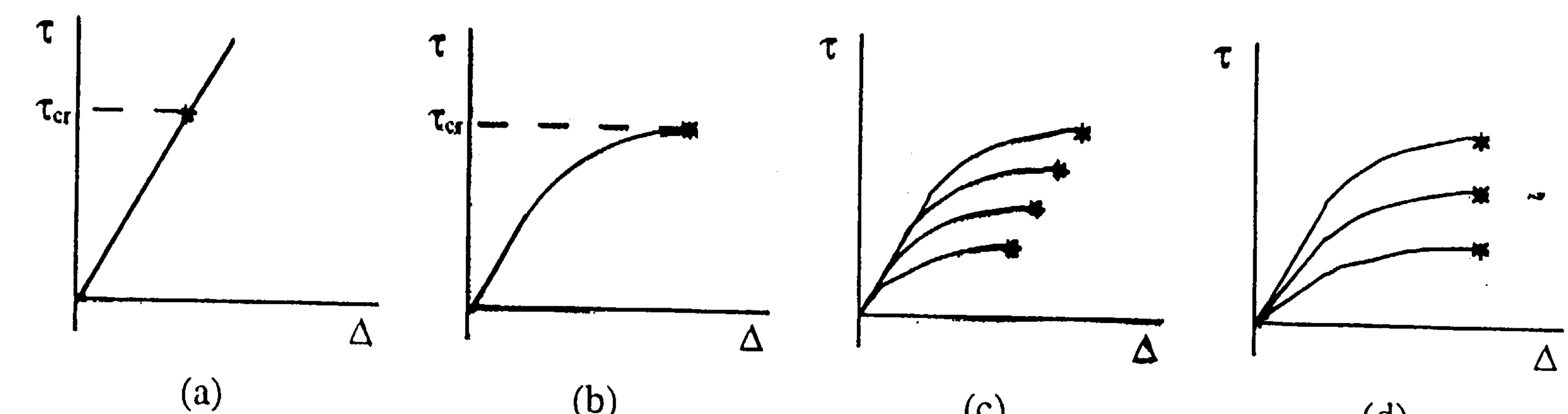
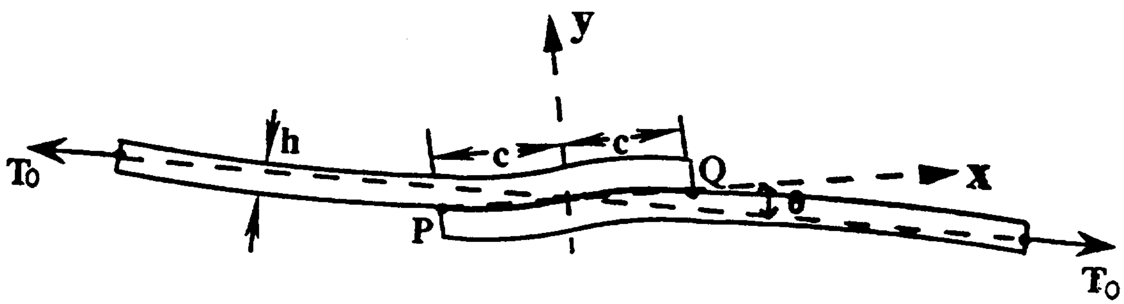


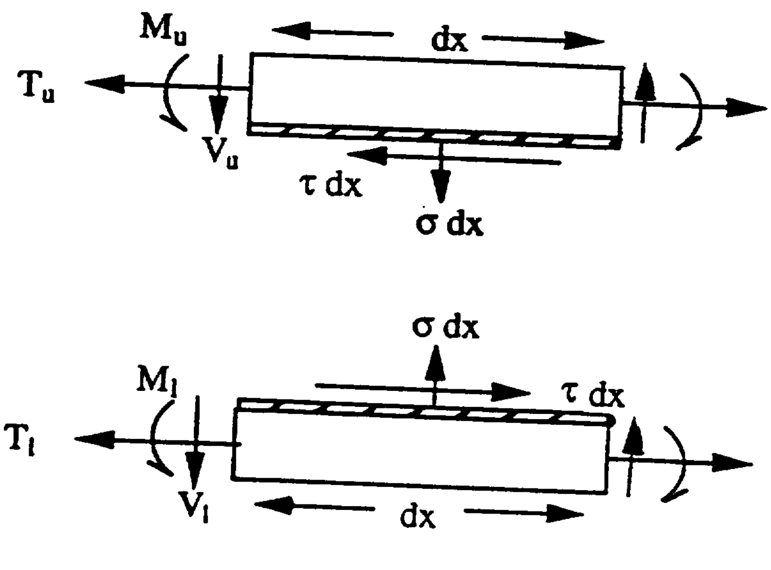
Figure 1. Traction-displacement curves and associated failure points.

Reference:

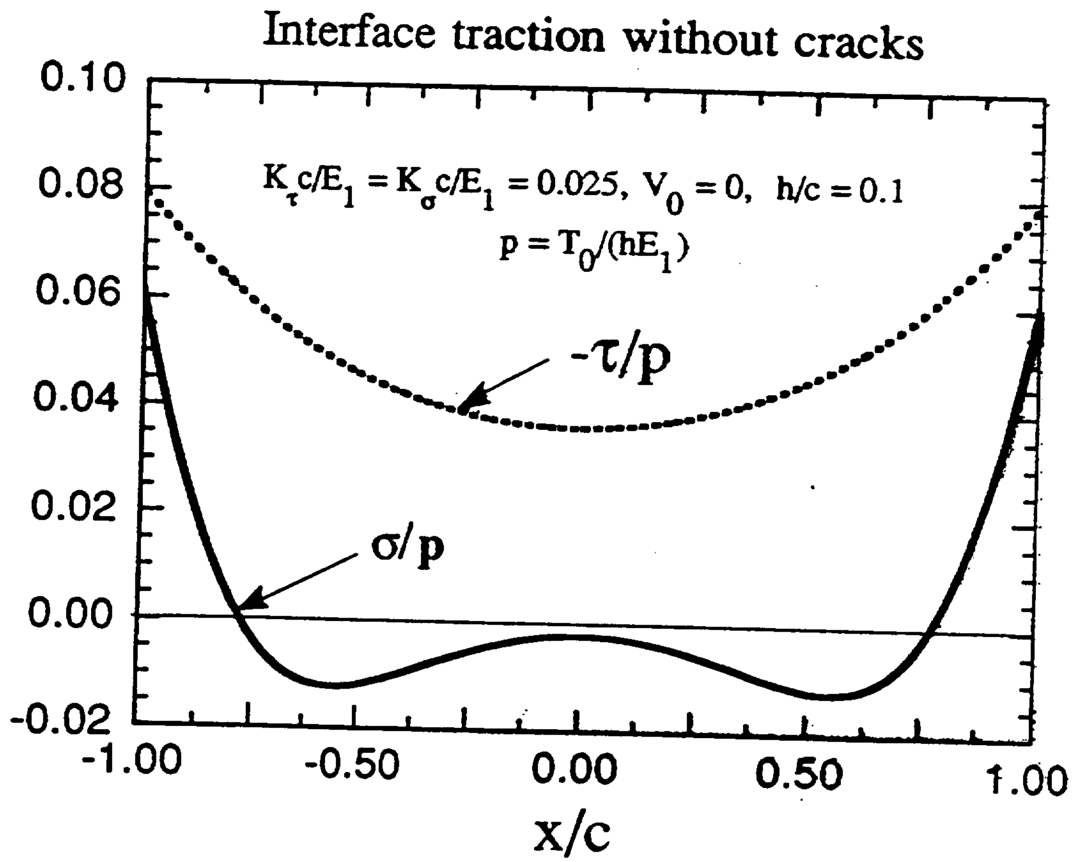
M. Goland and E. Reissner, "The Stresses in Cemented Joints", J. Appl. Mech, March 1944, ppA17-A27.



Replace adhesive layer by distributions of springs



$$\tau = K_\tau(u_u - u_l), \qquad \sigma = K_\sigma(w_u - w_l)$$

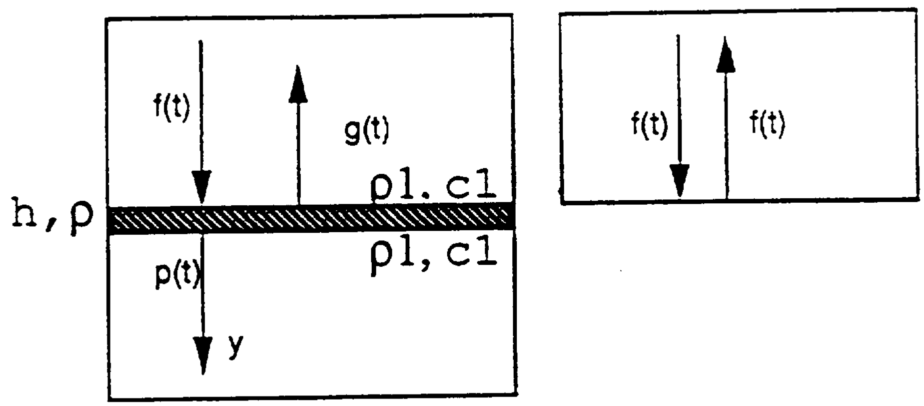


Interface traction without cracks
$K_\tau c/E_1 = K_\sigma c/E_1 = 0.025$, $V_0 = 0$, $h/c = 0.1$
$p = T_0/(hE_1)$
$-\tau/p$
σ/p
0.10
0.08
0.06
0.04
0.02
0.00
-0.02
-1.00
-0.50
0.00
0.50
1.00
x/c

OBJECTIVES

1. Relate residual strength of fatigue loaded adhesive bonds to initiation of a nonlinear relation between traction, τ, and extension, Δ.
2. Develop a theoretical model to relate the slope of the $\tau - \Delta$ curve to the ultrasonic signals reflected from bonds.
3. Formulate the inverse problem.
4. Supplement theory with experimental results.

THEORETICAL MODEL



$$\sigma_y|_{y=0+} - \sigma_y|_{y=0-} = \frac{1}{2}\rho h[\ddot{\nu}|_{y=0+} + \ddot{\nu}|_{y=0-}] \quad (1)$$

$$\bar{\sigma}_y = \frac{1}{2}[\sigma_{y|y=0+} + \sigma_{y=0-}] \quad (2)$$

$$\delta = \nu|_{y=0+} - \nu|_{y=0-} \quad (3)$$

$$\bar{\sigma} = \beta\delta \quad (4)$$

$$\beta = \gamma\beta_0 \quad (5)$$

$$\nu^I(y,t) = e^{i(\omega t - ky)} \tag{6}$$

$$\nu^R(y,t) = Re^{i(\omega t + ky)} \tag{7}$$

$$\nu^T(y,t) = Te^{i(\omega t - ky)} \tag{8}$$

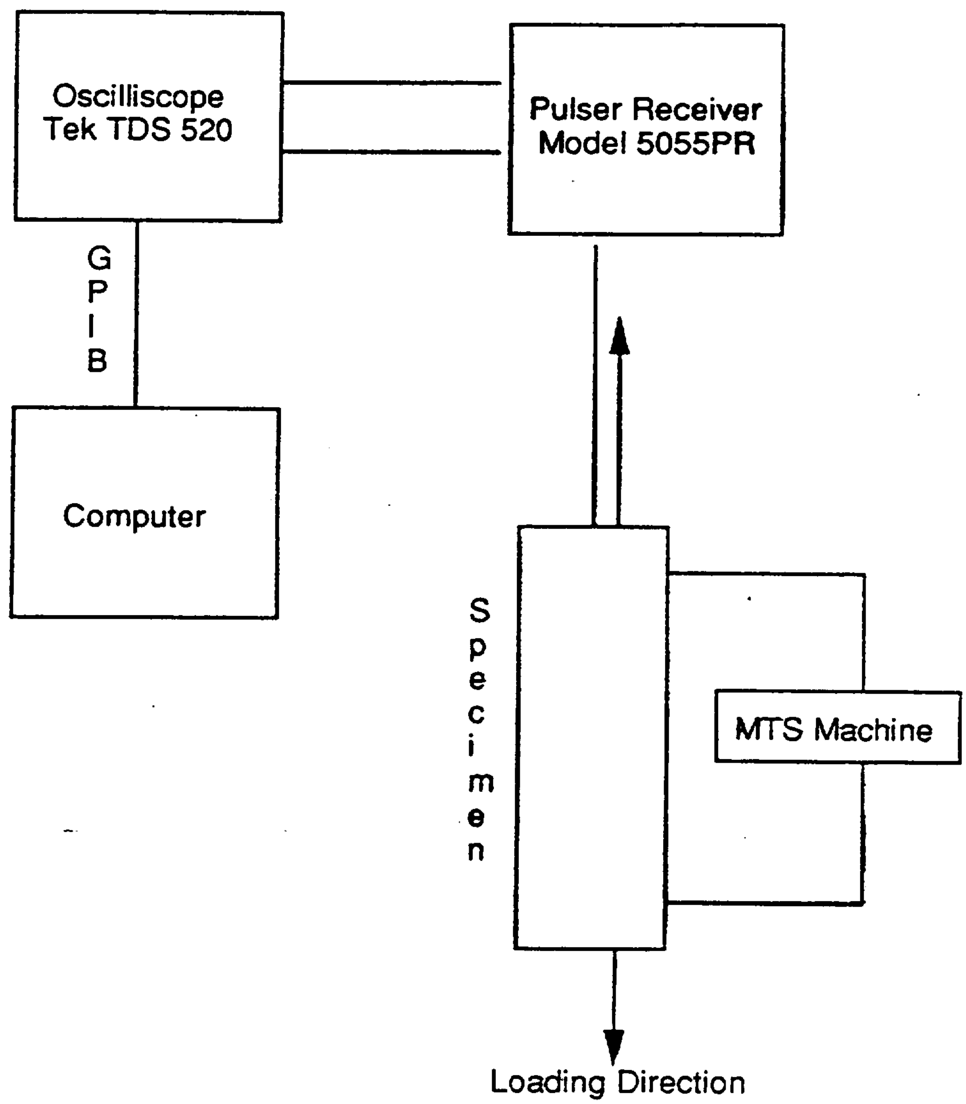
$$R(\omega) = \frac{i\bar{\omega}}{i\bar{\omega} + 2\gamma} - \frac{i\bar{\omega}}{i\bar{\omega} + 1} \tag{9}$$

where $\bar{\omega} = \frac{\rho h}{2\rho_1 c_1}\omega$

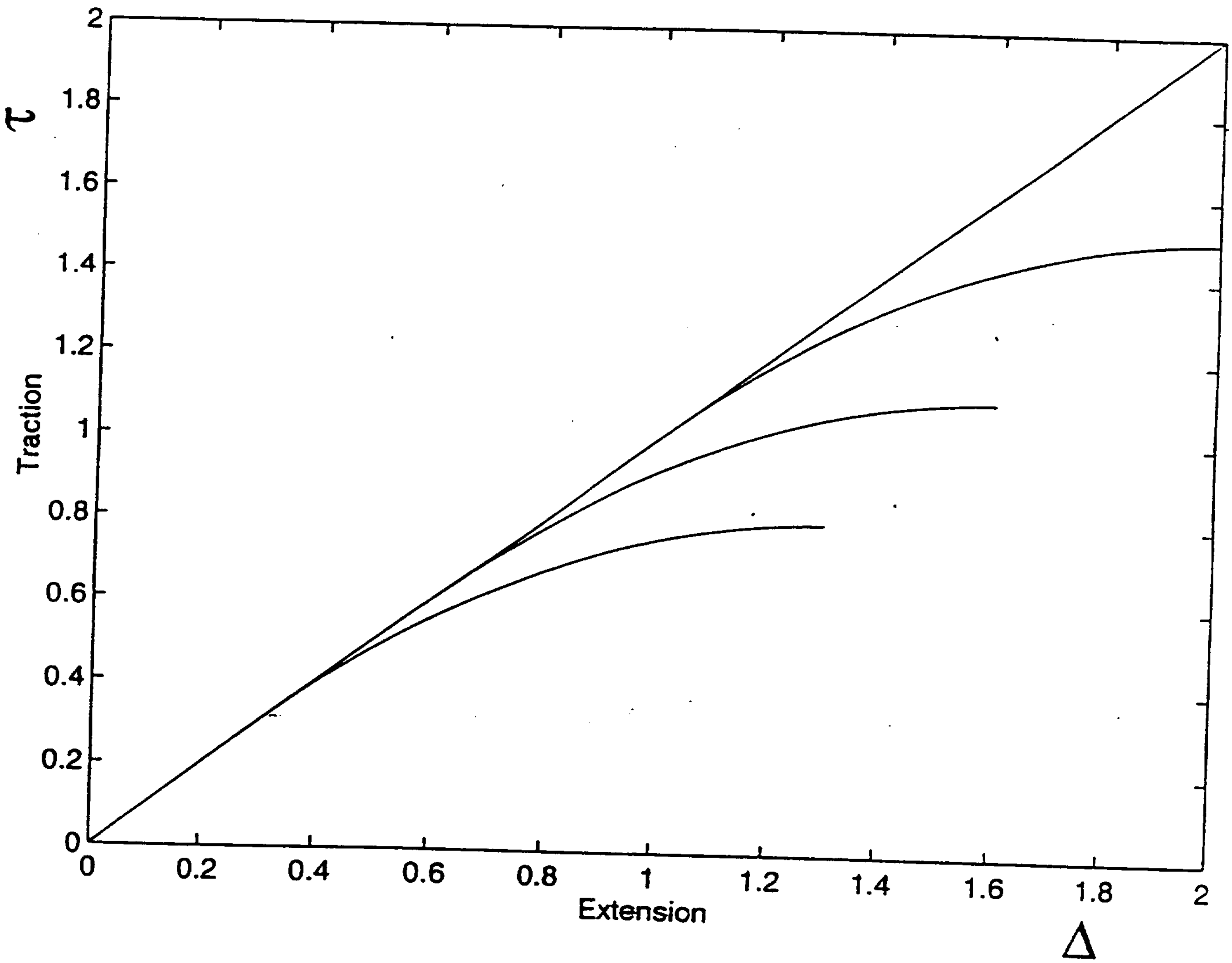
$$g^s(t) = \int_{-\infty}^{\infty} R(\omega)F(\omega)e^{-i\omega t}\,d\omega \tag{10}$$

$$Err(\gamma) = \sum_{i=1}^{N}(g(t_i) - g^s(t_i,\gamma))^2 \tag{11}$$

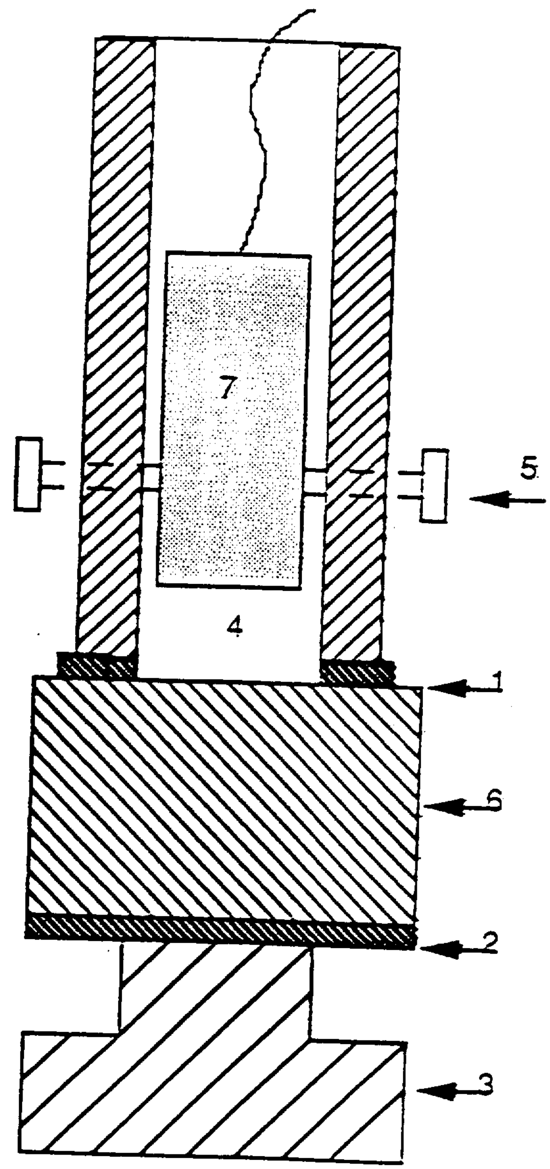
EXPERIMENT SETUP



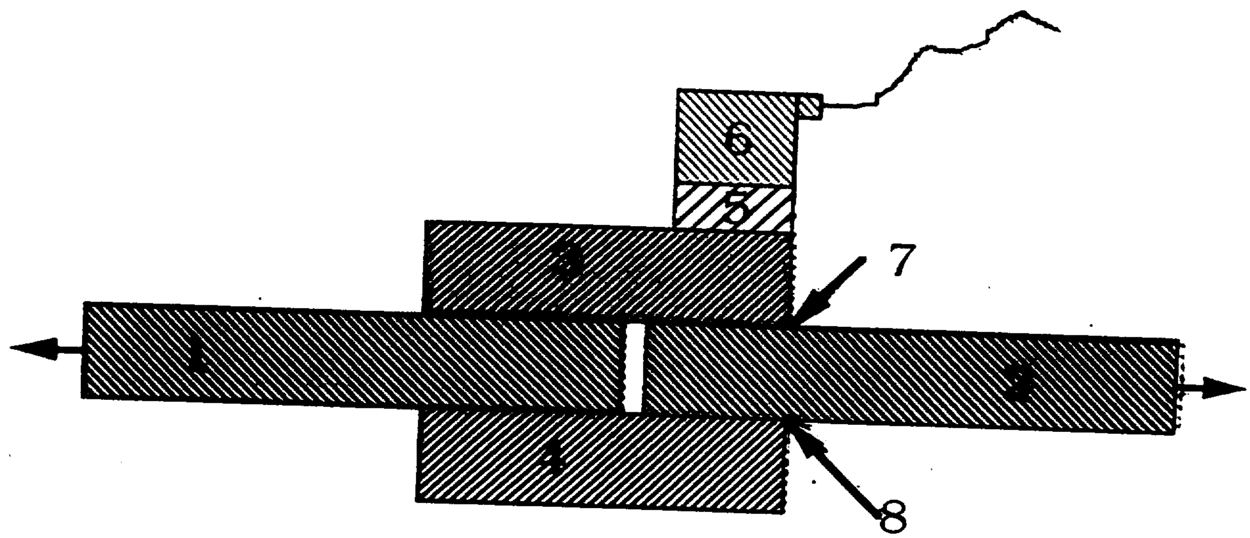
TYPICAL NONLINEAR ELASTIC $\tau - \Delta$ RELATION



SPECIMEN



1. Adhesive (connection)
2. adhesive (testing layer)
3. Al block (adherend 2)
4. Al Tube (water tank)
5. Screw
6. Al block (adherend 1)
7. Transducer



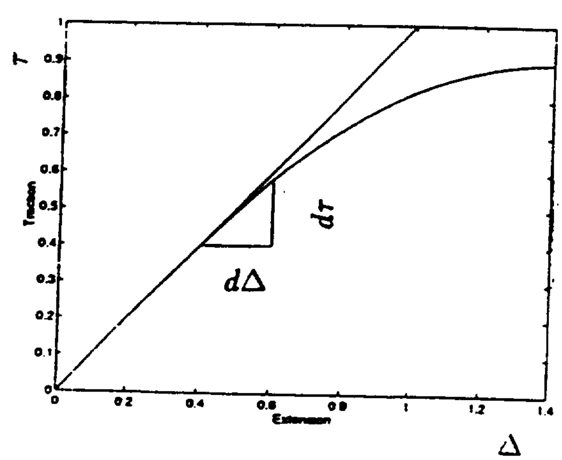
1. Aluminum piece a
2. Aluminum piece b
3. Strip a
4. Strip b
5. Delay block
6. Shear wave transducer
7. Adhesive layer a
8. Adhesive layer b

Methodology of Nonlinear Behavior Study

Use different fatigue cycles to generate different severities of degradation.

By varying the static load, ultrasonic measurements allow us to get the slope of the $\tau - \Delta$ curve at several points.

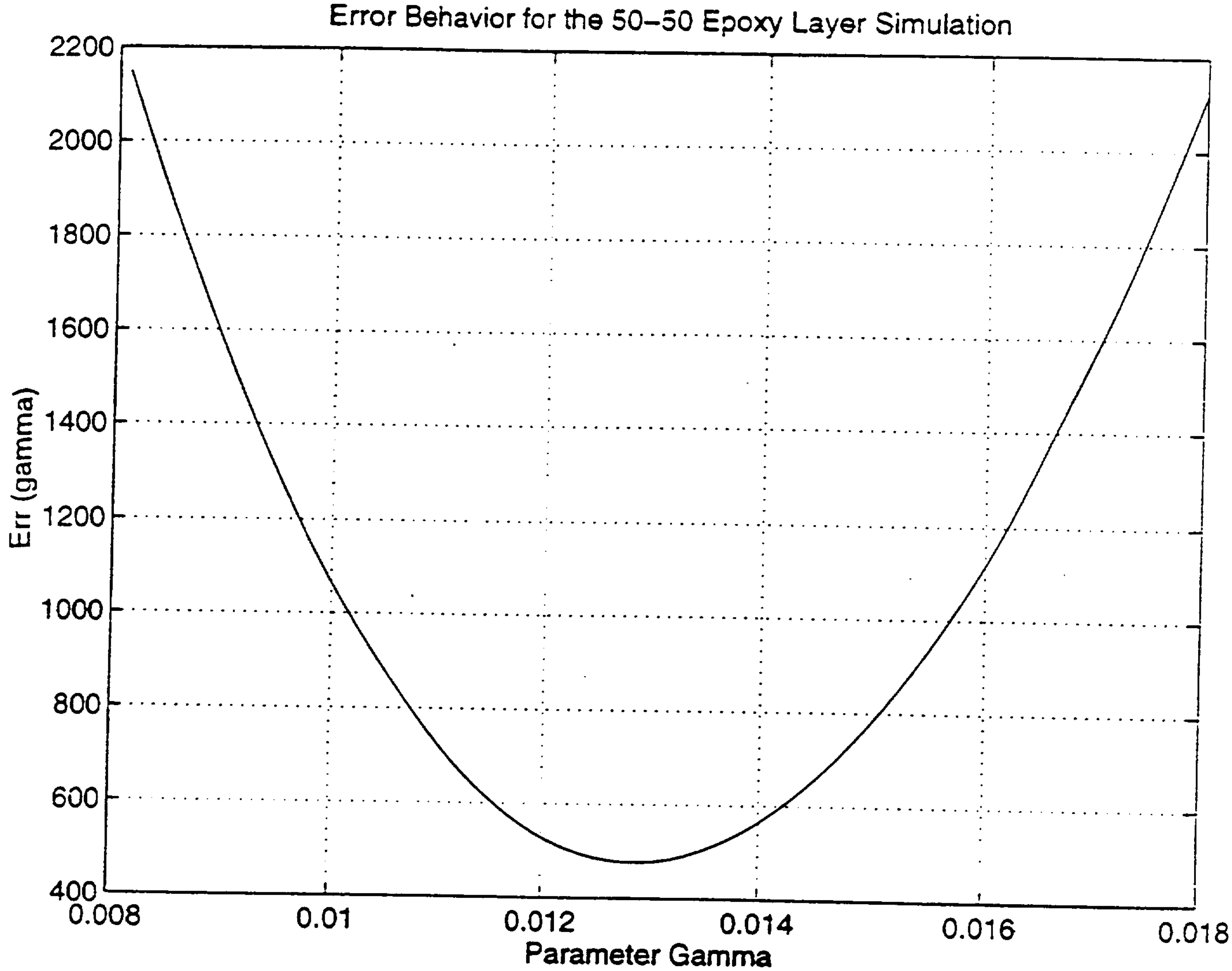
$$\frac{d\tau}{d\Delta} \approx \frac{\sigma}{\delta} = \beta$$



MODEL VERIFICATION

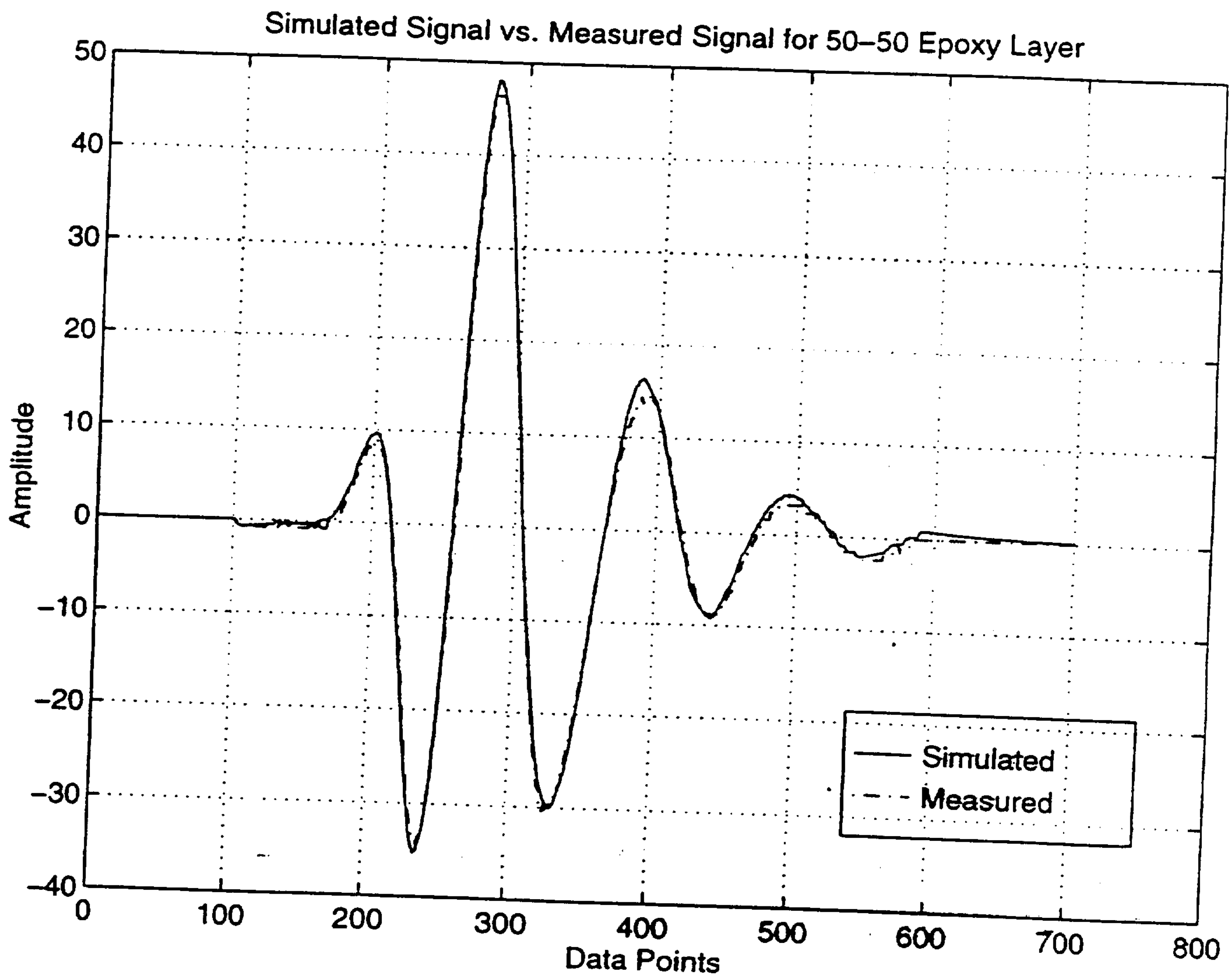
Epoxy Layer	Inversed Effective Modulus $\beta h (GPa)$	Calculated Lame Constants $\lambda + 2\mu (GPa)$
70-30	8.04	8.01
50-50	6.83	6.50

Error Function Behavior

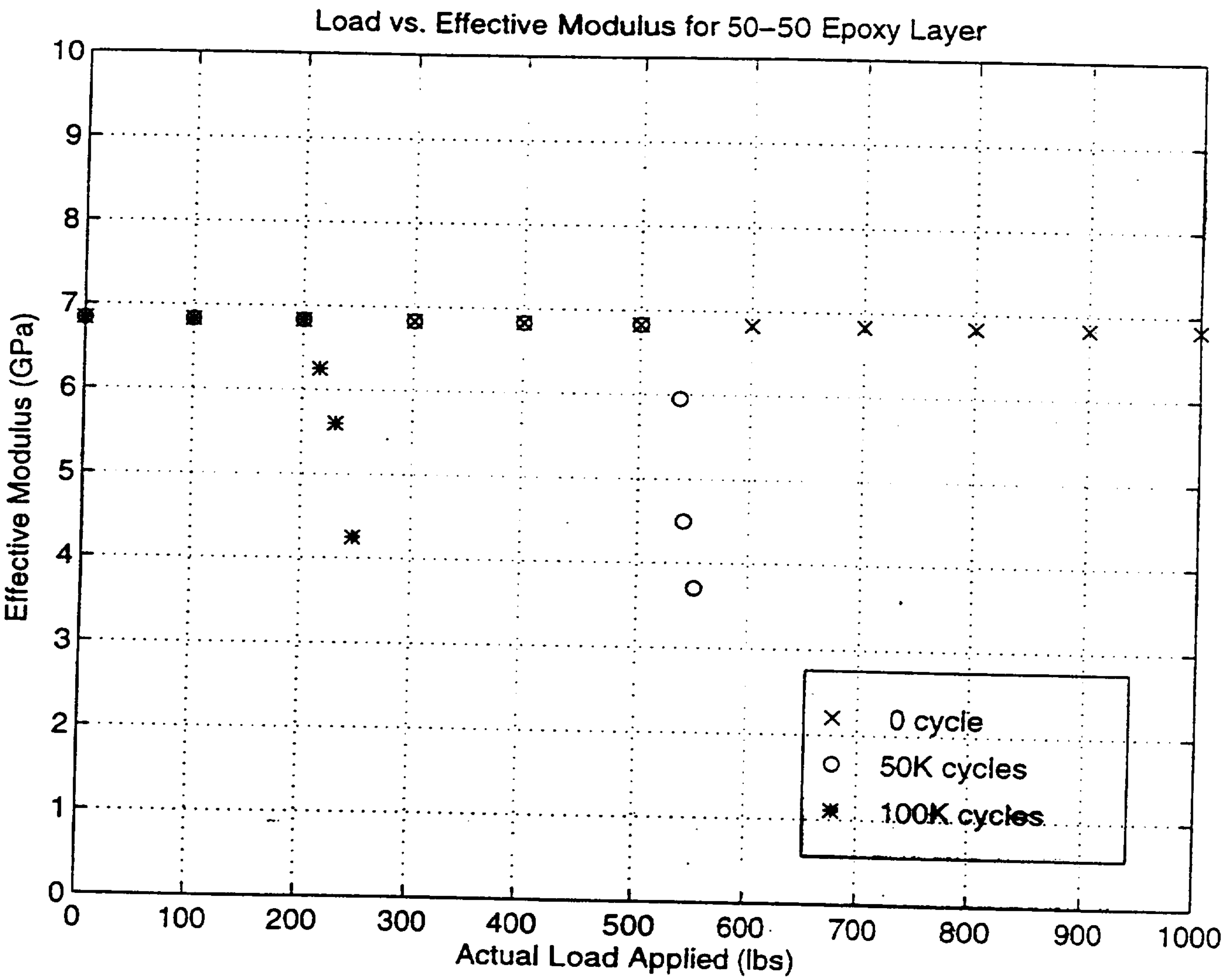


$$Err(\gamma) = \sum_{i=1}^{N} (g(t_i) - g^s(t_i, \gamma))^2$$

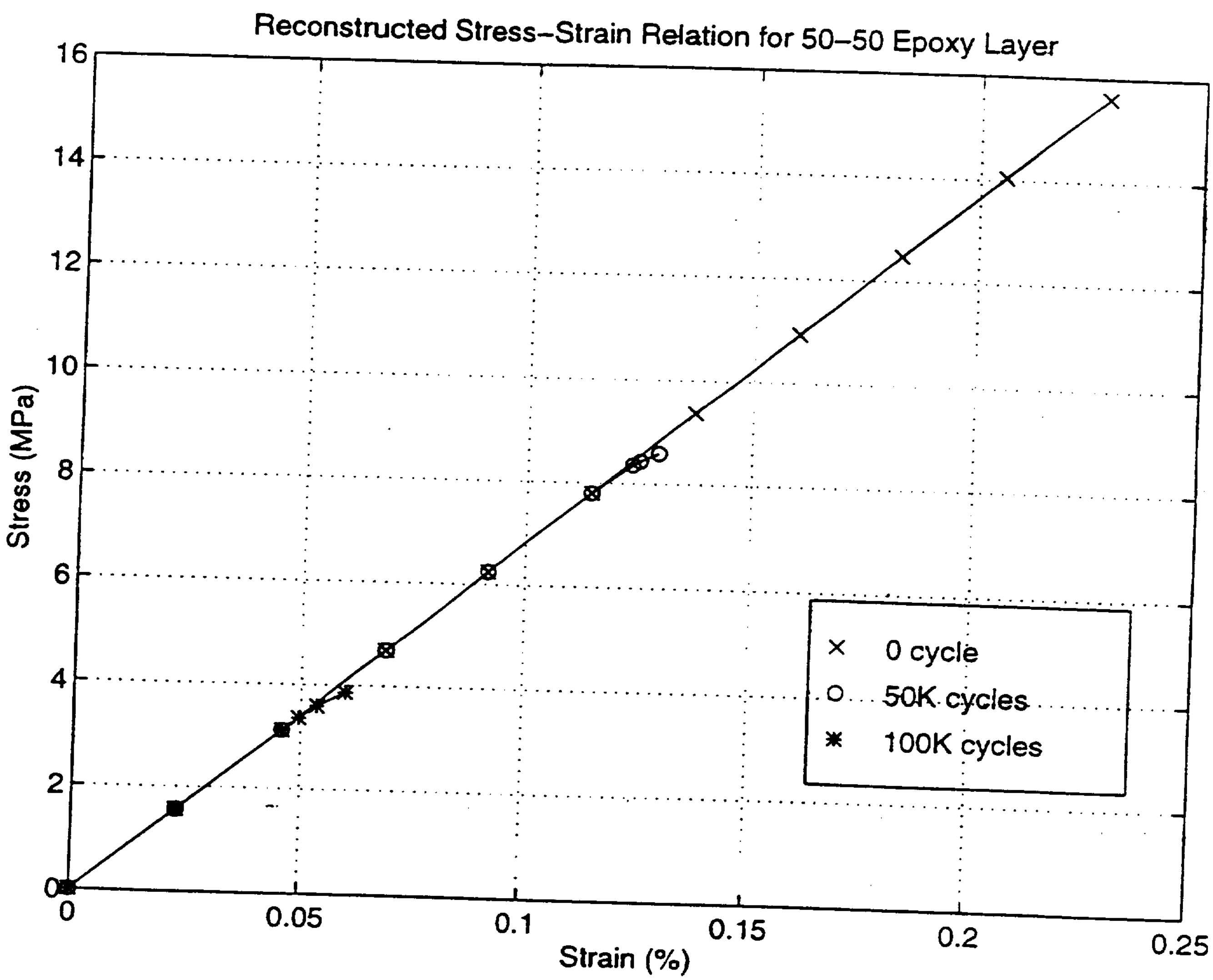
Simulated Signal vs. Measured Signal



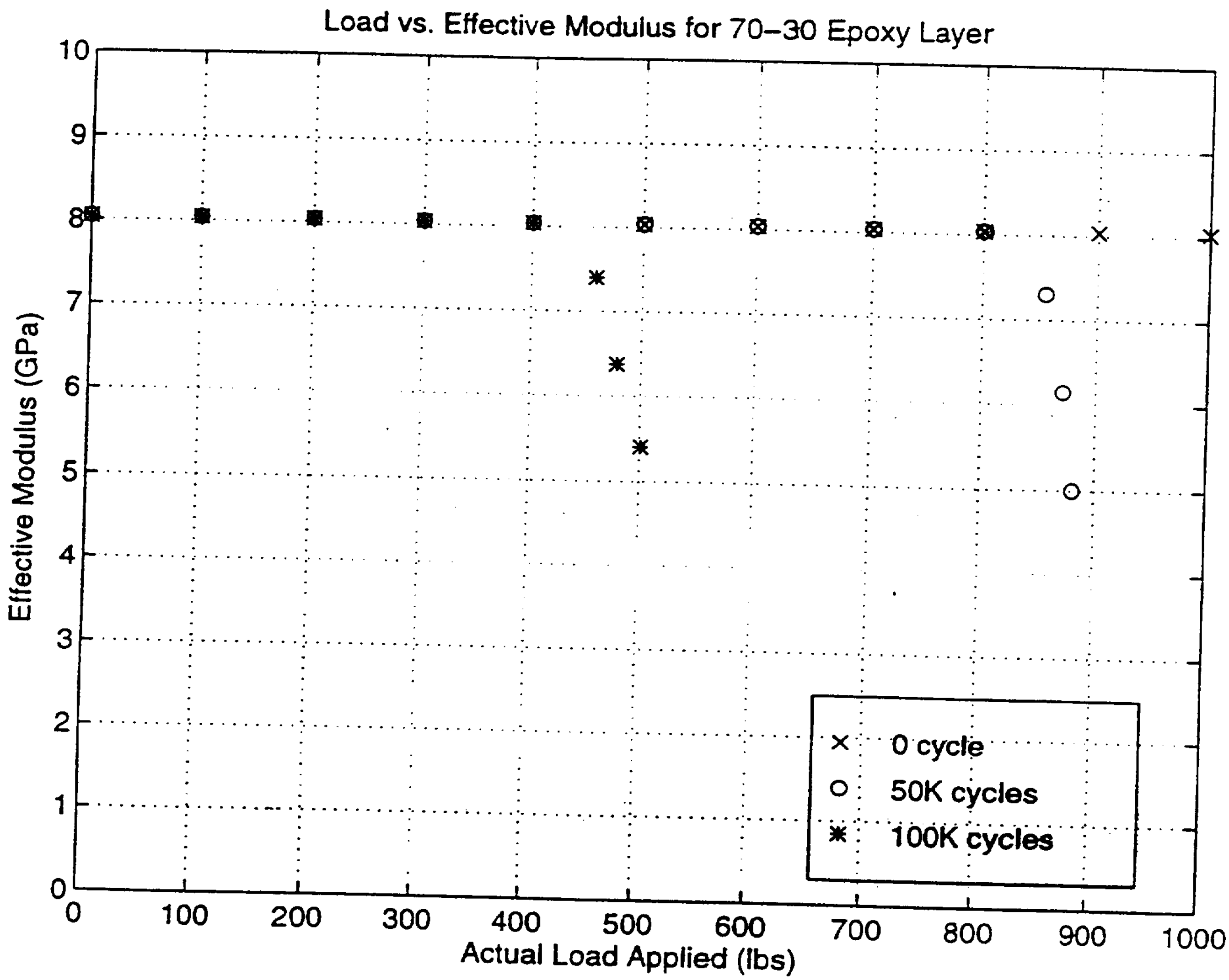
Load vs. Effective Modulus for 50-50 Epoxy Layer



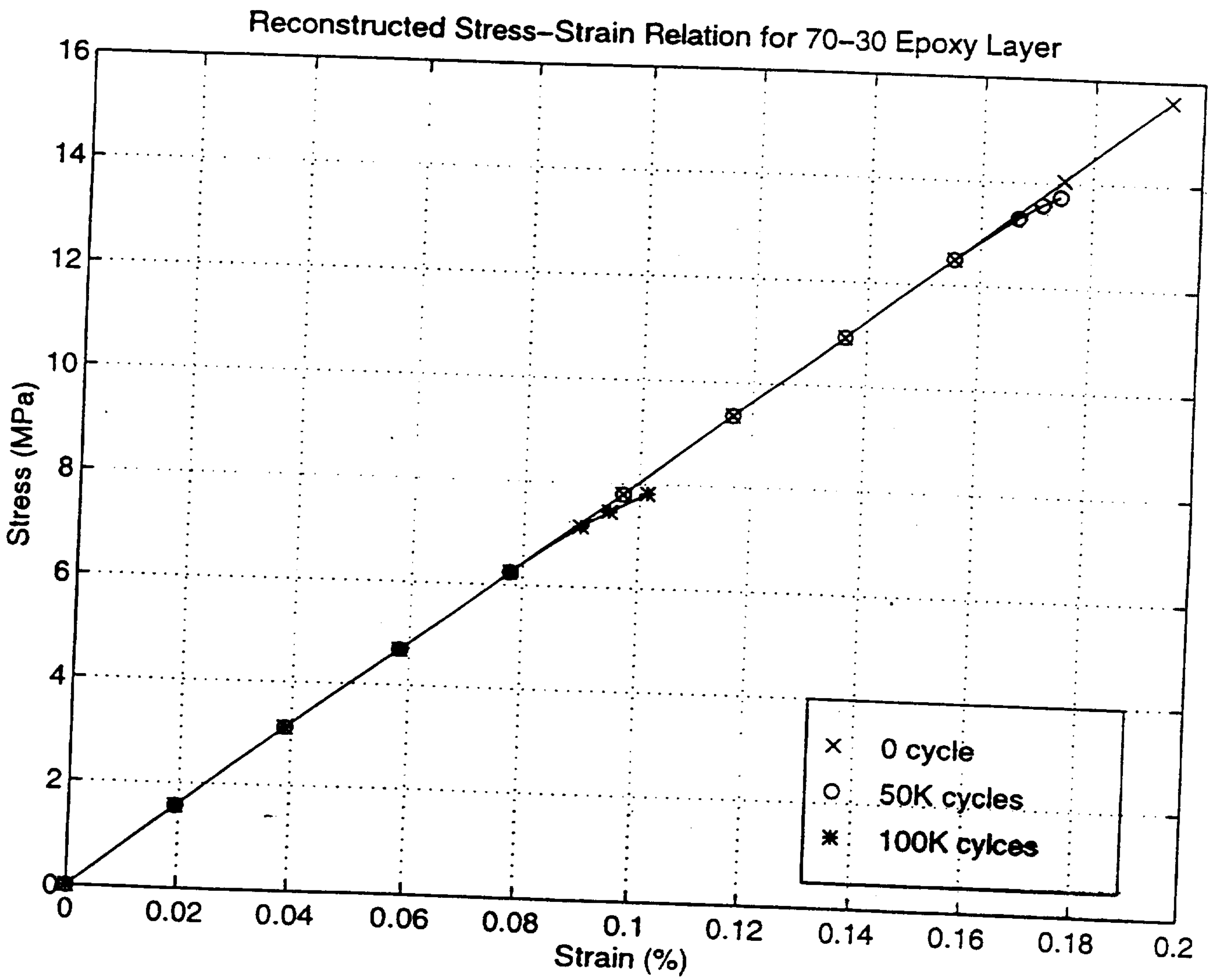
Reconstructed Stress-Strain Relation (50-50)



Load vs. Effective Modulus for 70-30 Epoxy Layer



Reconstructed Stress-Strain Relation (70-30)



CONCLUSIONS

The inverse problem based on the theoretical model yields the effective modulus of the adhesive layer.

The results show that the nonlinear behavior related to degradation due to cyclic fatigue can be detected by the reduction of the linear portion of the stress-strain curve without any change of slope in the linear range.

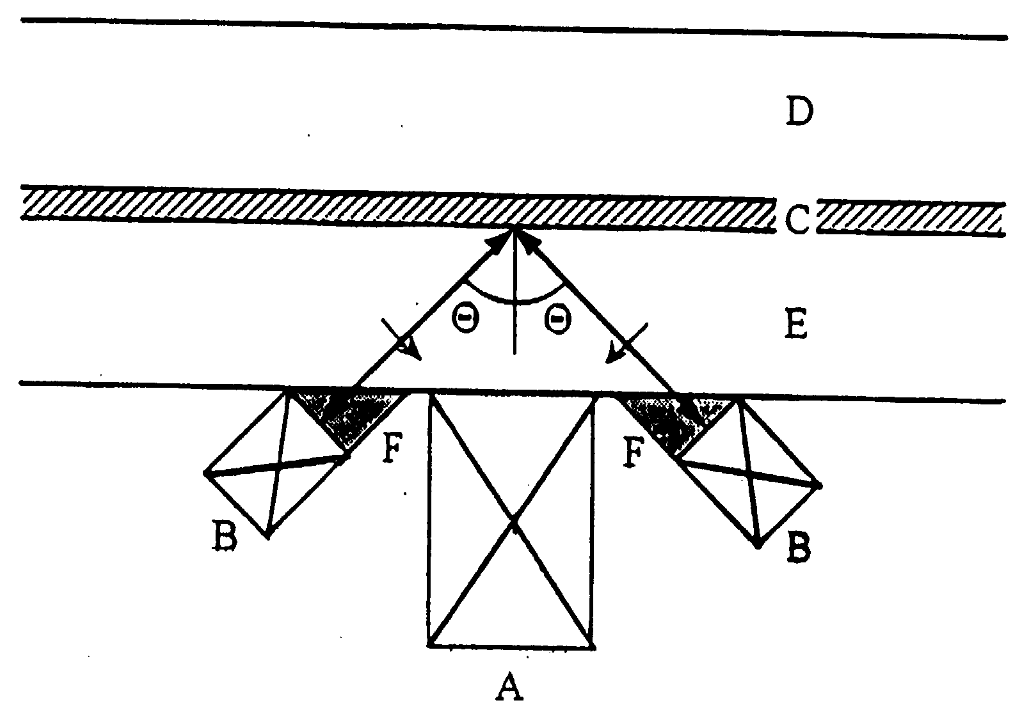


Figure 2. Schematic diagram of one possible experimental arrangement showing (A) low-frequency standing-wave generating transducer, (B) probe transverse wave transducers (with polarization as shown), (C) adhesive layer, (D) adherend 1, (E) adherend 2, and (F) buffer blocks for probe beam coupling.

CURRENT WORK (2nd year)

1. Load adhesive bond in shear in MTS machine.
2. Ultrasonic test with shear waves.
3. Shear-fatigue adhesive bond.
4. Use ultrasound to detect onset of nonlinearity.

FUTURE WORK (3rd year)

Apply shear load using low-frequency ultrasound or low-frequency electromagnetic transducer.

Nondestructive Determination of Bond Strength

Tobias P. Berndt and Robert E. Green, Jr.

CNDE

Center for Nondestructive Evaluation

The Johns Hopkins University

Department of Materials Science and Engineering

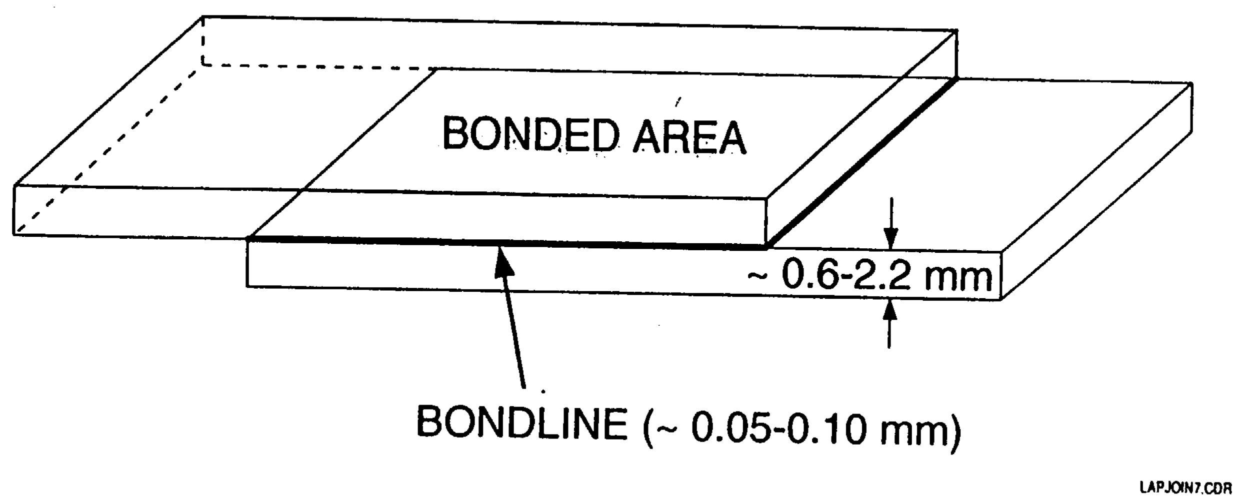
Acknowledgments:

Funding by NASA Langley and CNDE

Samples provided by BOEING

November 4, 1997

Adhesively Bonded Lap Joint Sample



Proposed Techniques

Linear Ultrasonic Waves

➤ **Nonlinear Ultrasonic Waves**

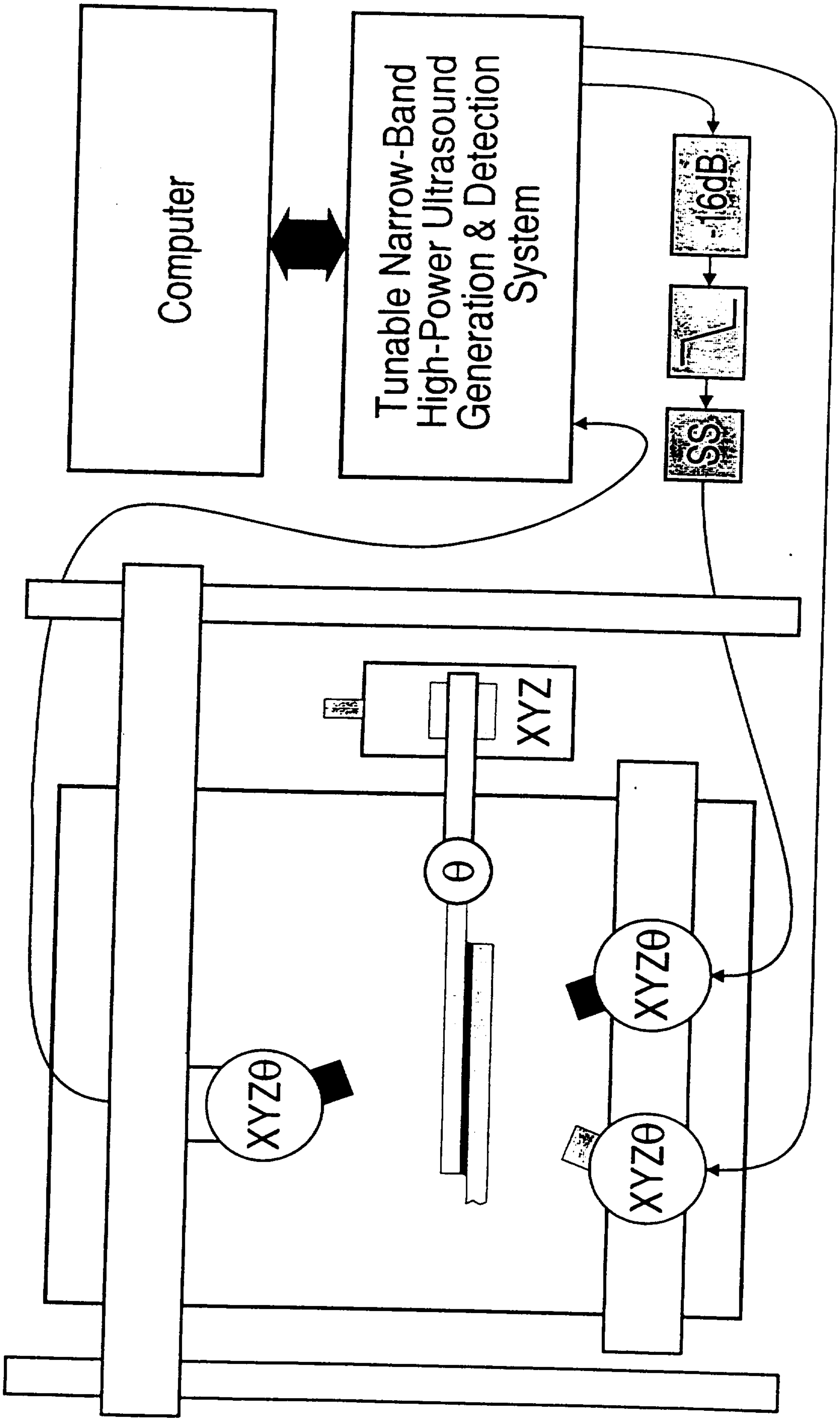
Acoustic Emission

Acousto-Ultrasonics

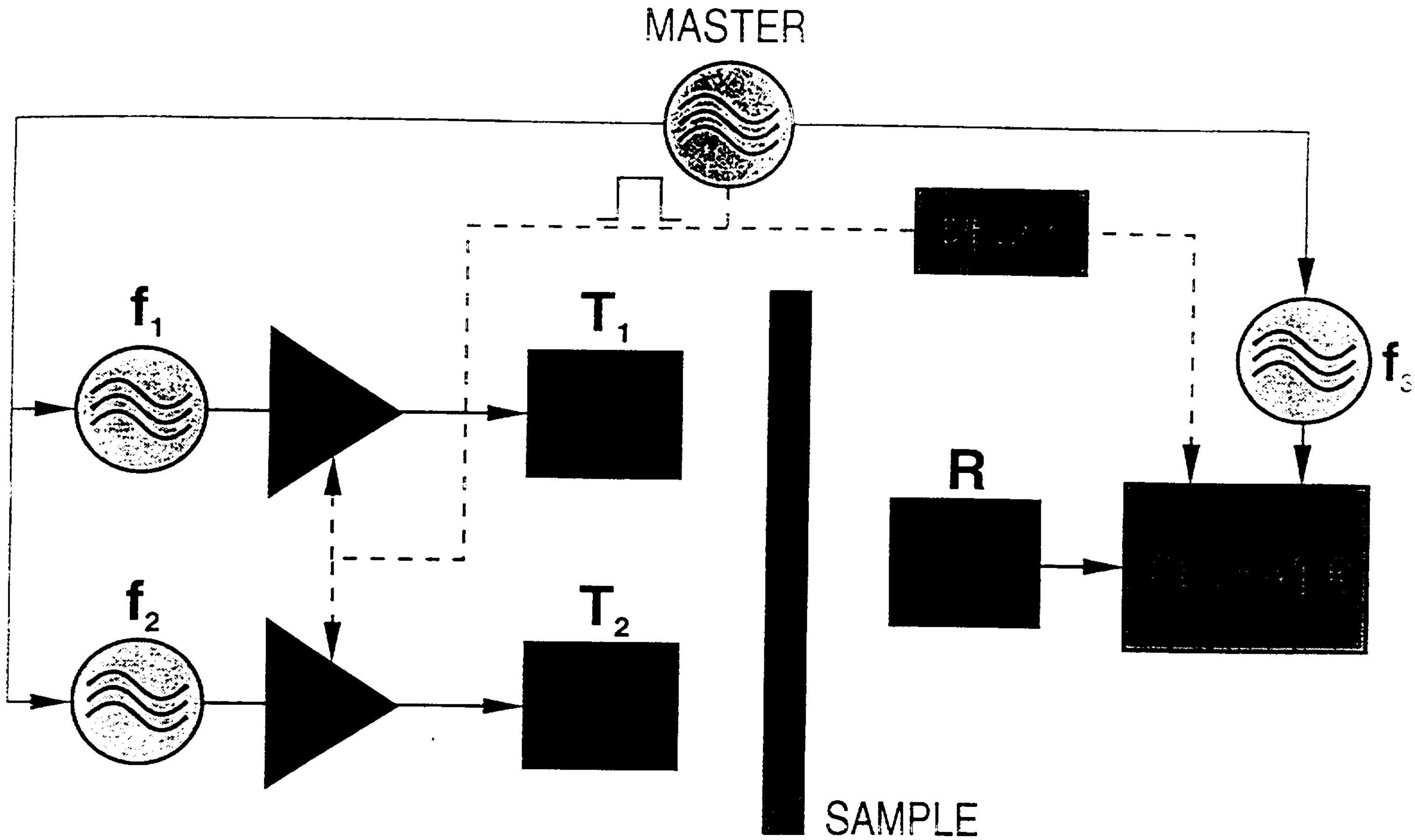
Non-Contact Ultrasonics

Tap Testing

Vibrational Techniques



EXPSET06.CDR



$$f_3 = N * f_1$$

$$f_3 = N * f_2$$

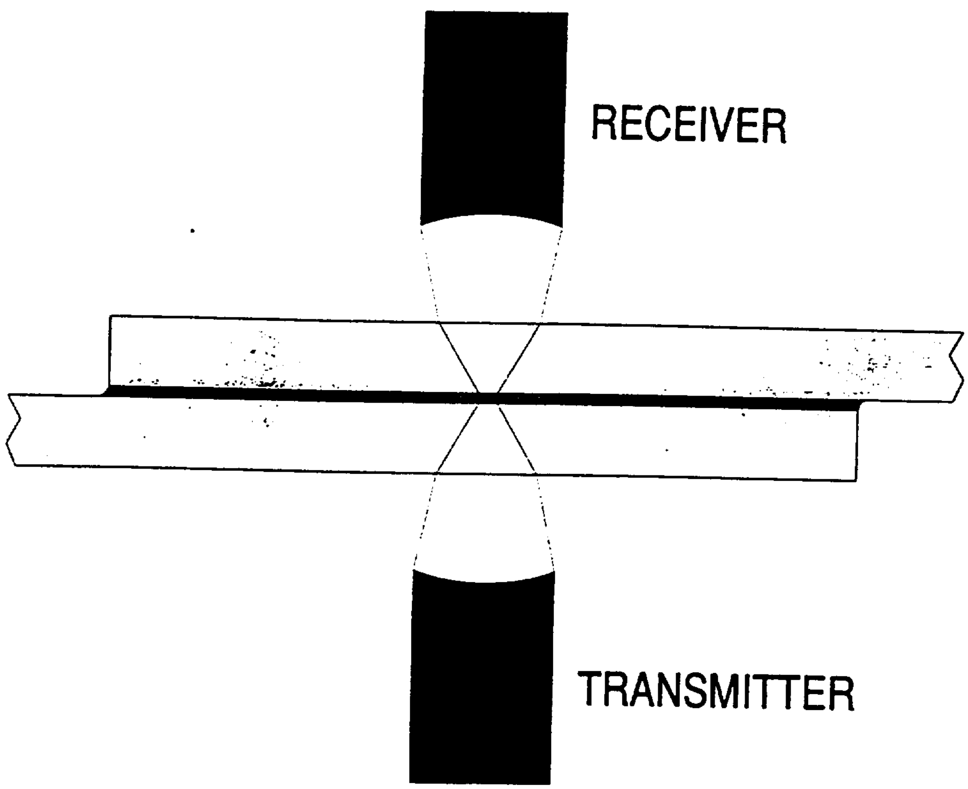
$$f_3 = f_1 + f_2$$

$$f_3 = f_1 - f_2$$

EXPSET04.CDR

Single Beam Interaction

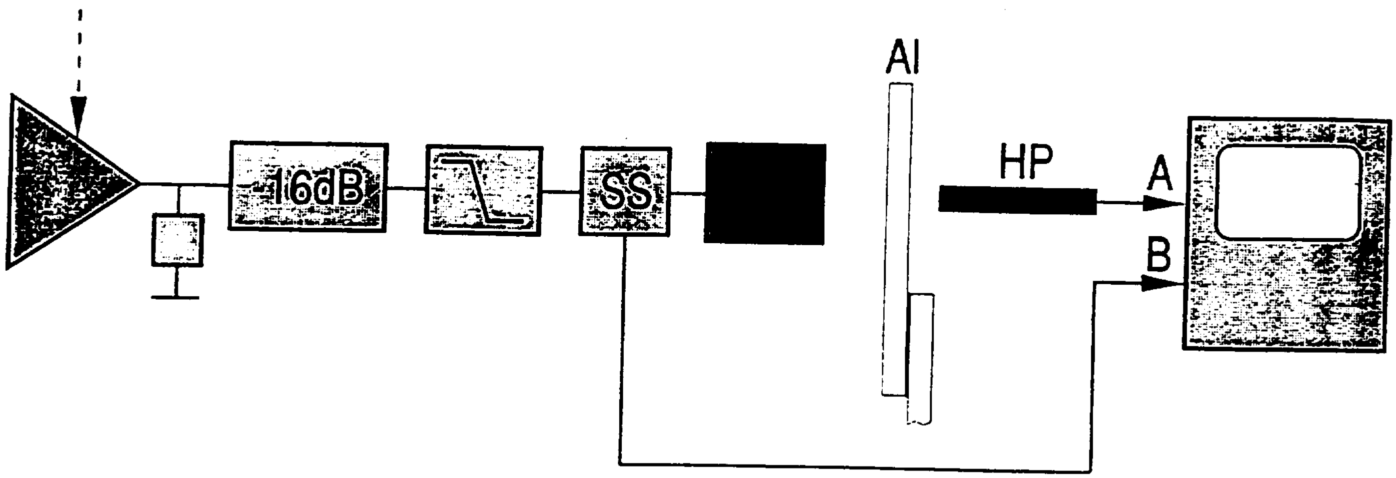
a) - Longitudinal Transmission

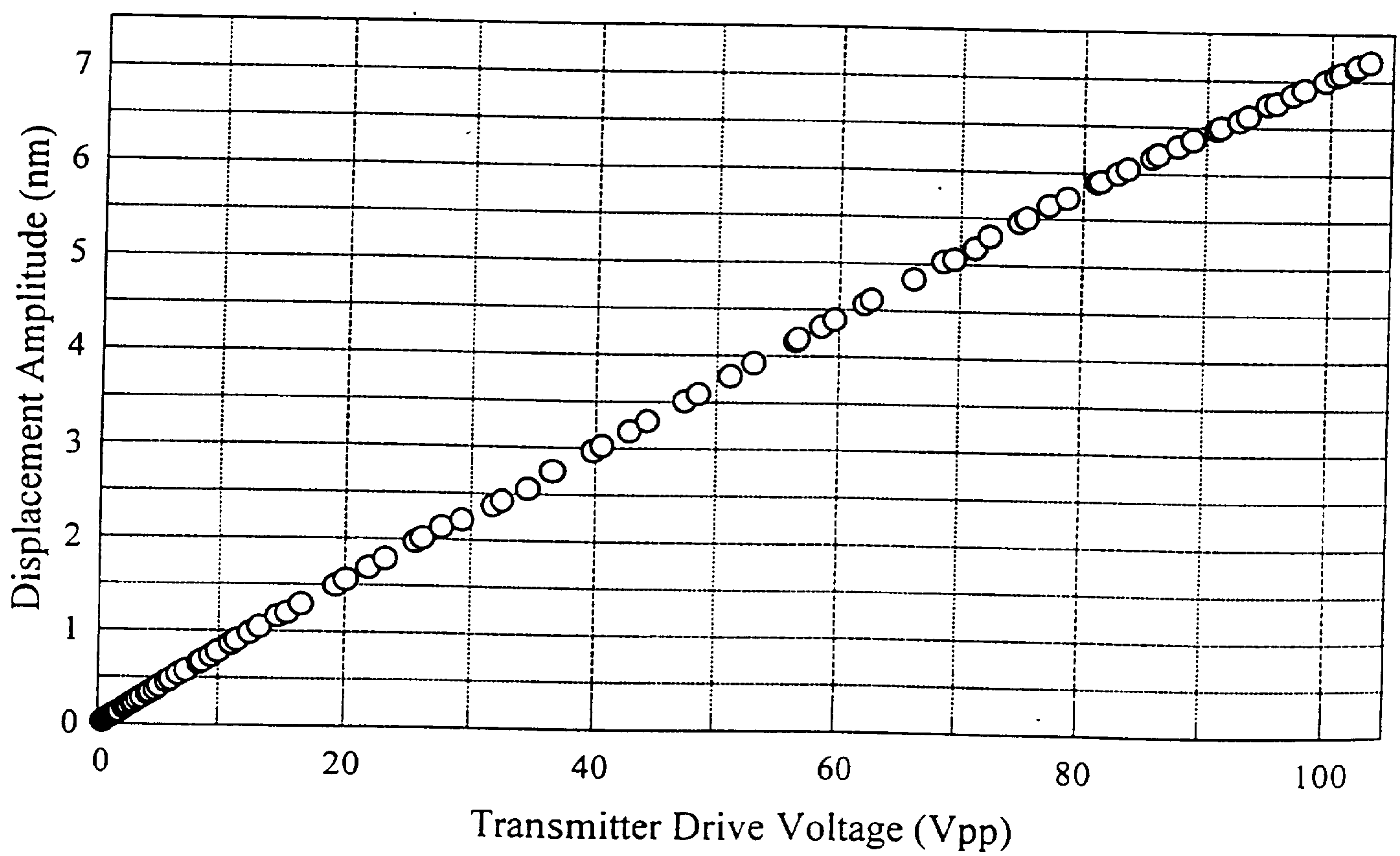


SFLTT_3.CDR

Nonlinear Parameter of Bond Sample #10 C-Scan

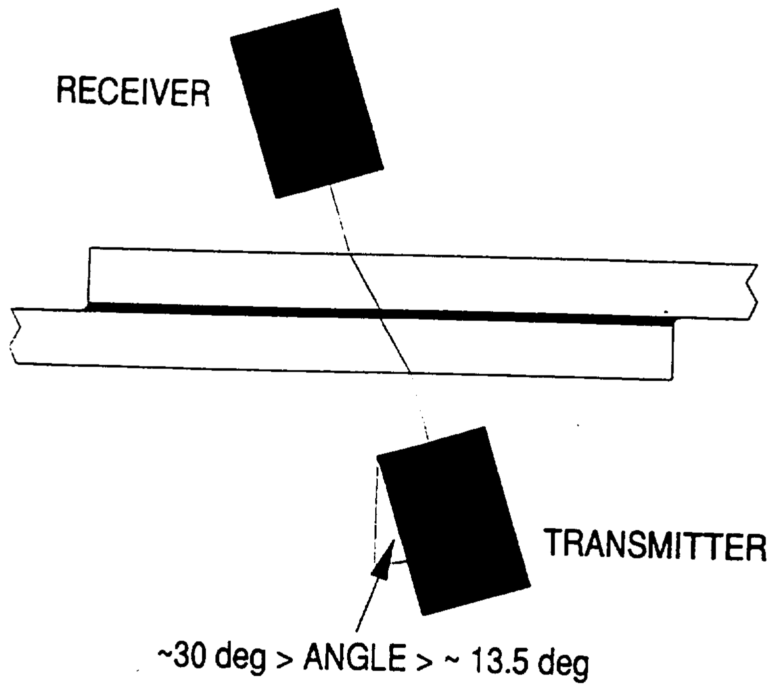
Surface Displacement as a Function of Input Powe



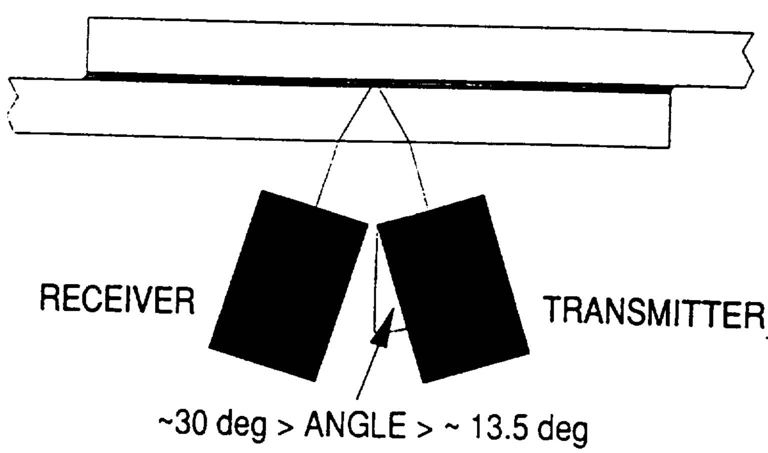


Single Beam Interaction

b) - Mode Converted Shear Transmission

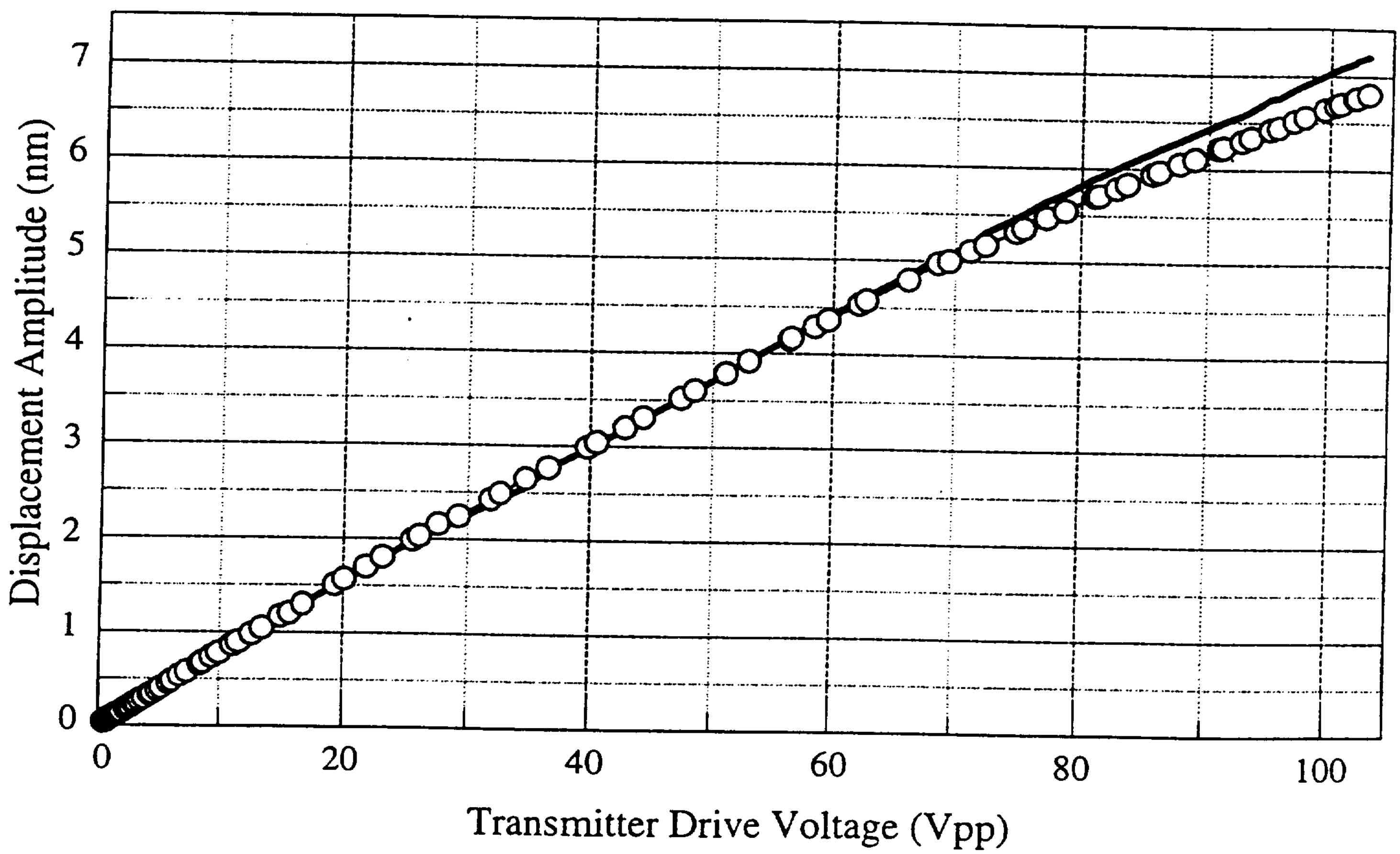


c) - Mode Converted Shear Reflection

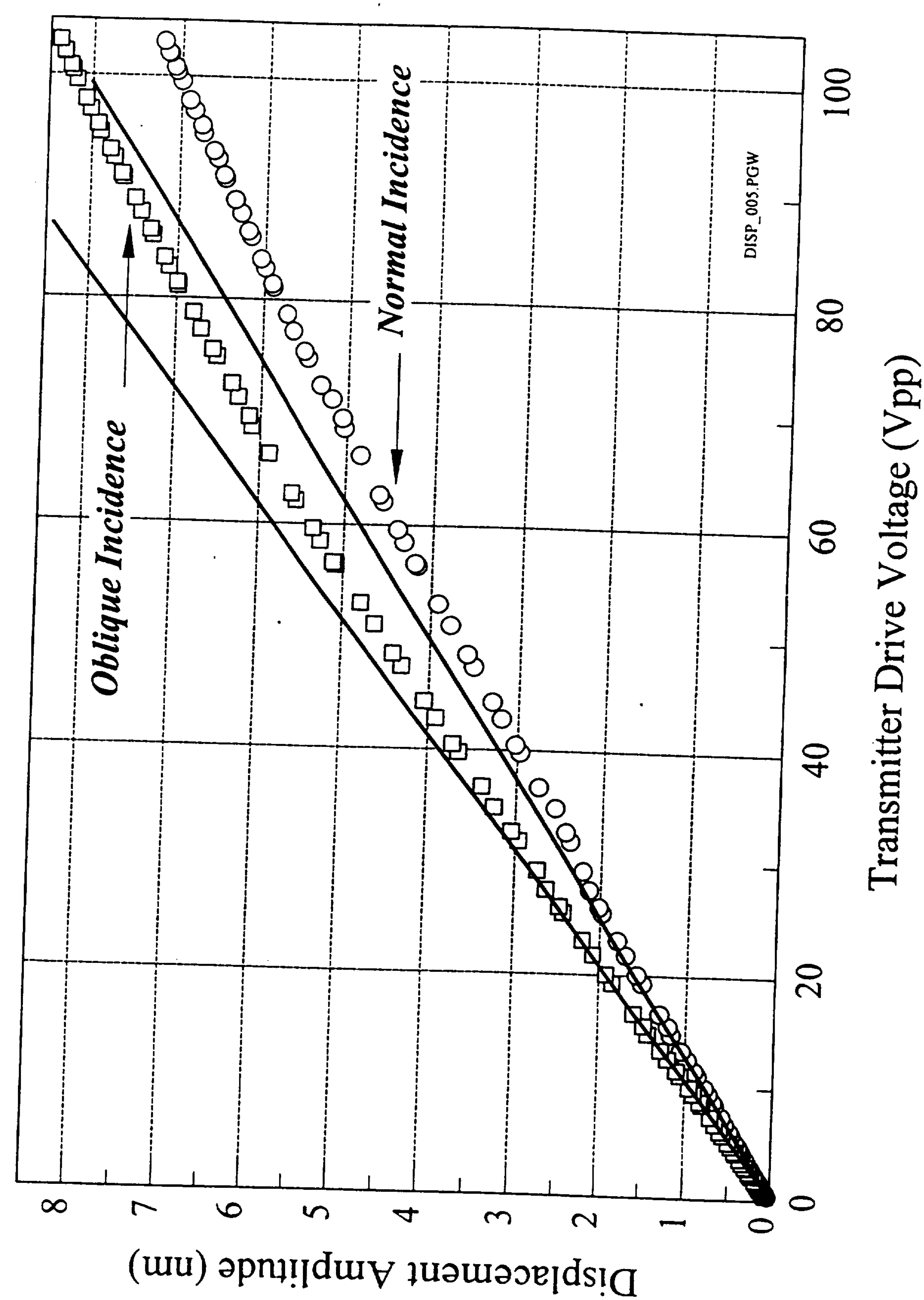


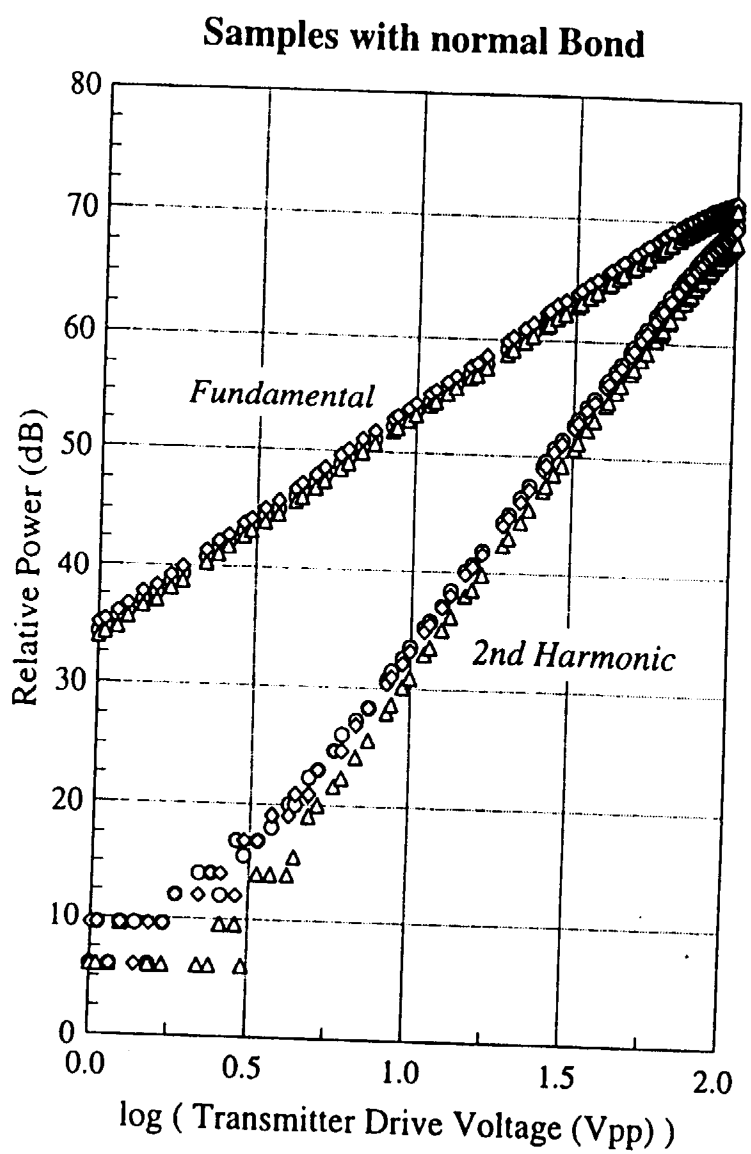
SFSREFL2.CDR

Displacement behind tilted (17.5°) Al-plate

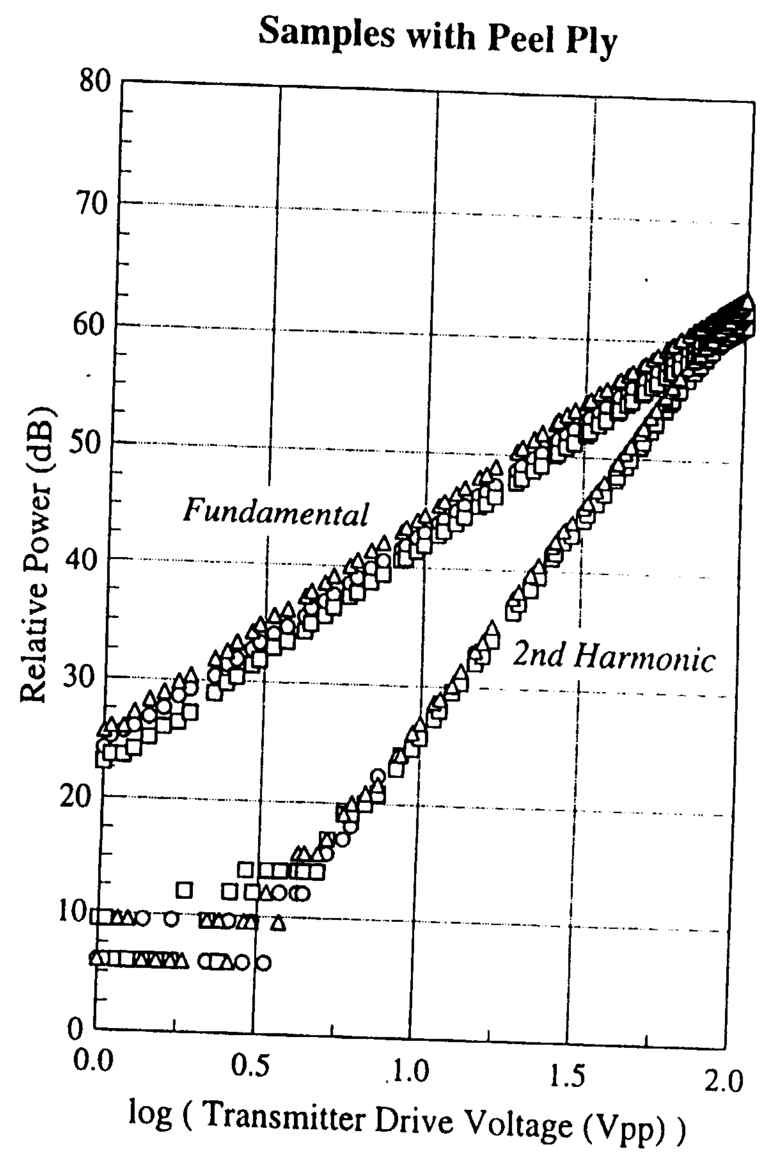


DISPCAL2.PGW


Displacement Amplitude (nm)
Transmitter Drive Voltage (Vpp)
Oblique Incidence
Normal Incidence
DISP_005.PGW
0
1
2
3
4
5
6
7
8
0
20
40
60
80
100

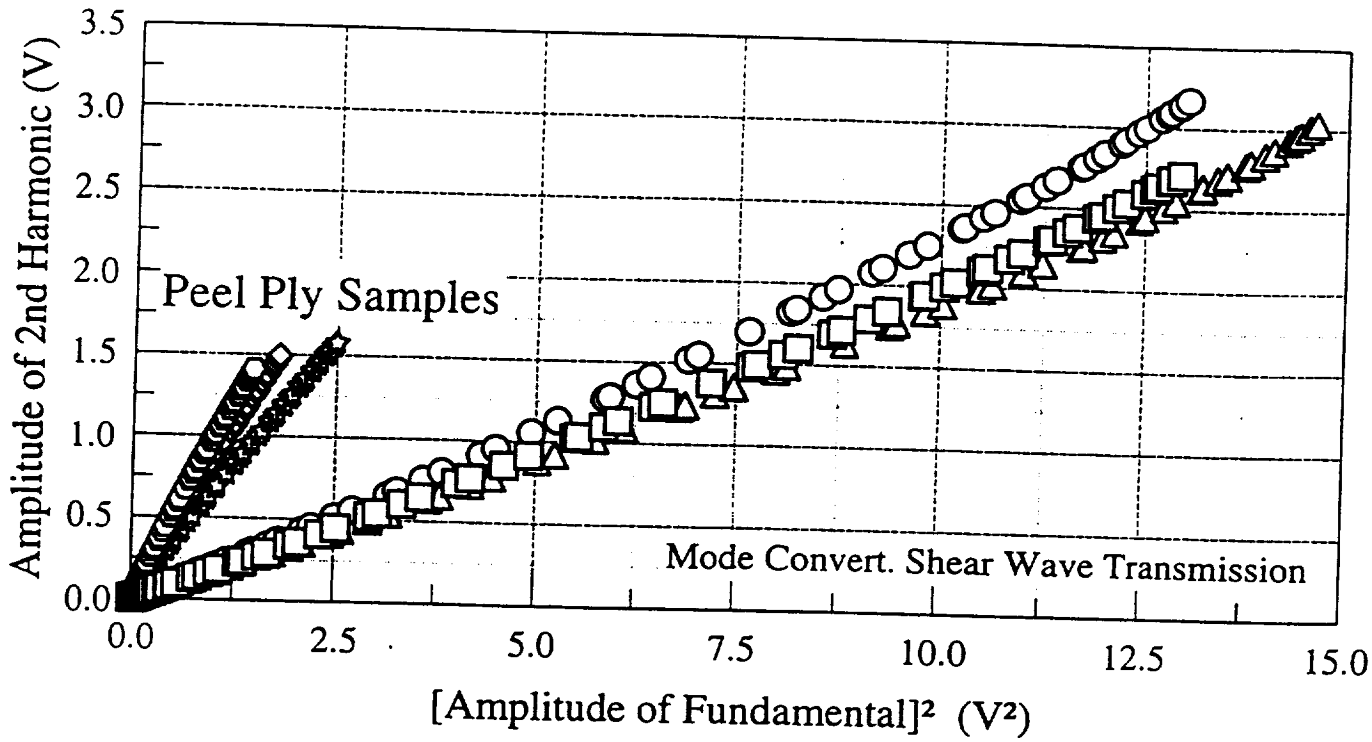

Samples with normal Bond
Relative Power (dB)
80
70
60
50
40
30
20
10
0
Fundamental
2nd Harmonic
0.0
0.5
1.0
1.5
2.0
log (Transmitter Drive Voltage (Vpp))

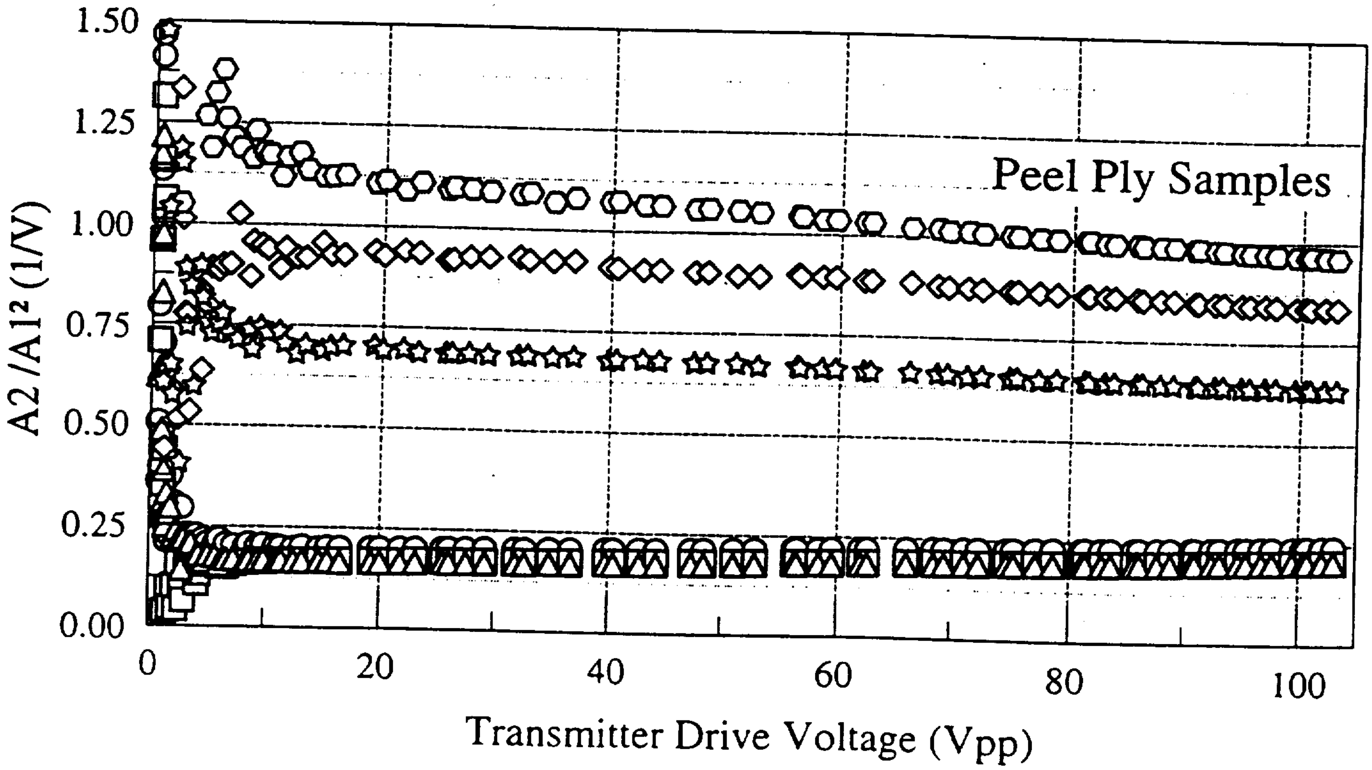
(a)


Samples with Peel Ply
Relative Power (dB)
80
70
60
50
40
30
20
10
0
Fundamental
2nd Harmonic
0.0
0.5
1.0
1.5
2.0
log (Transmitter Drive Voltage (Vpp))

(b)

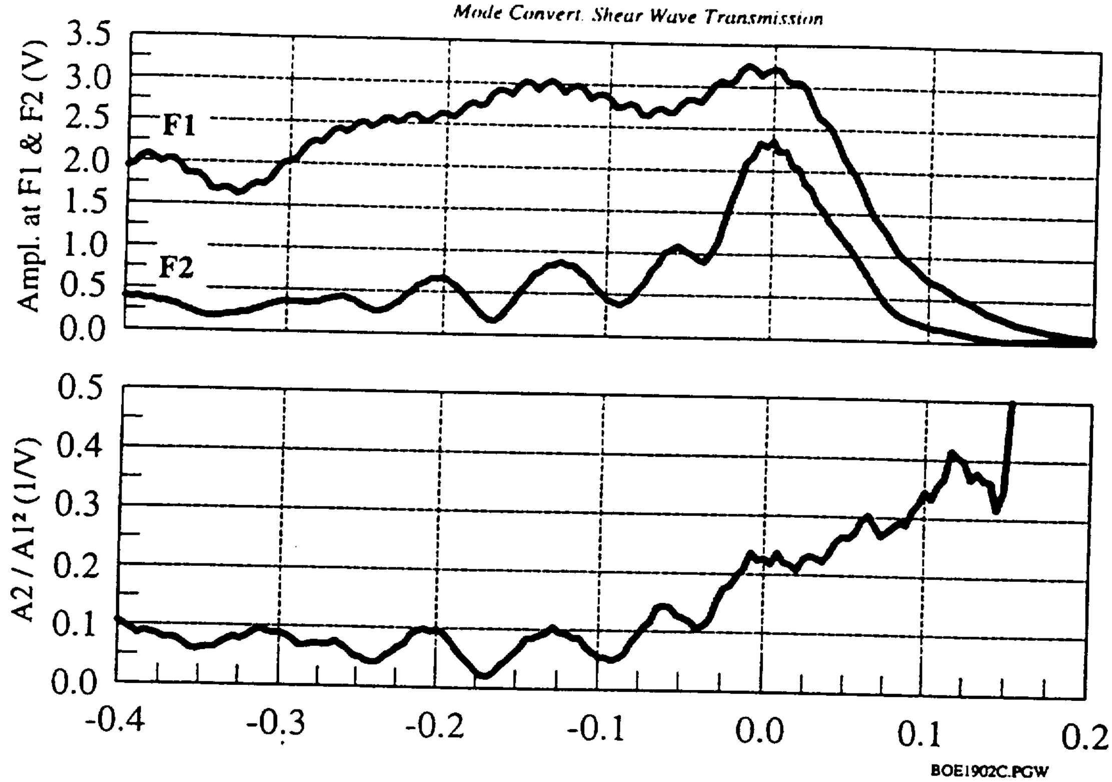
Comparison of all BOEING Samples



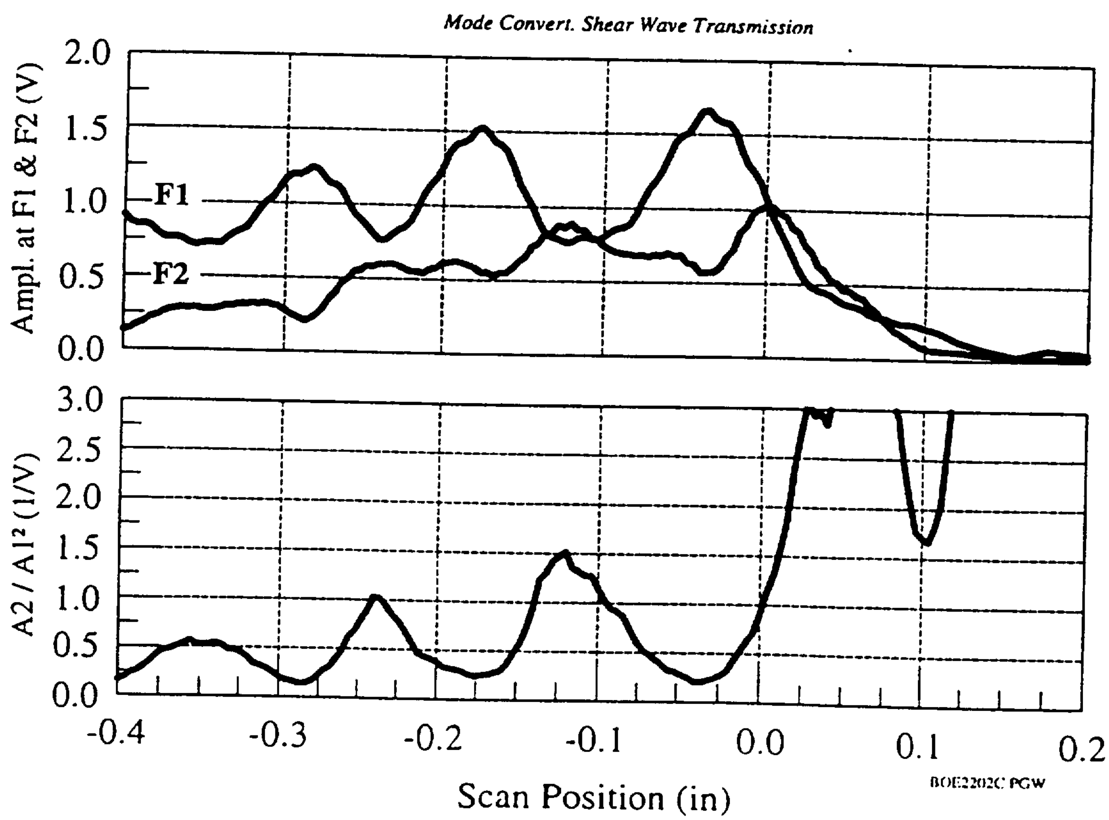


BOE_NLPc.PGW

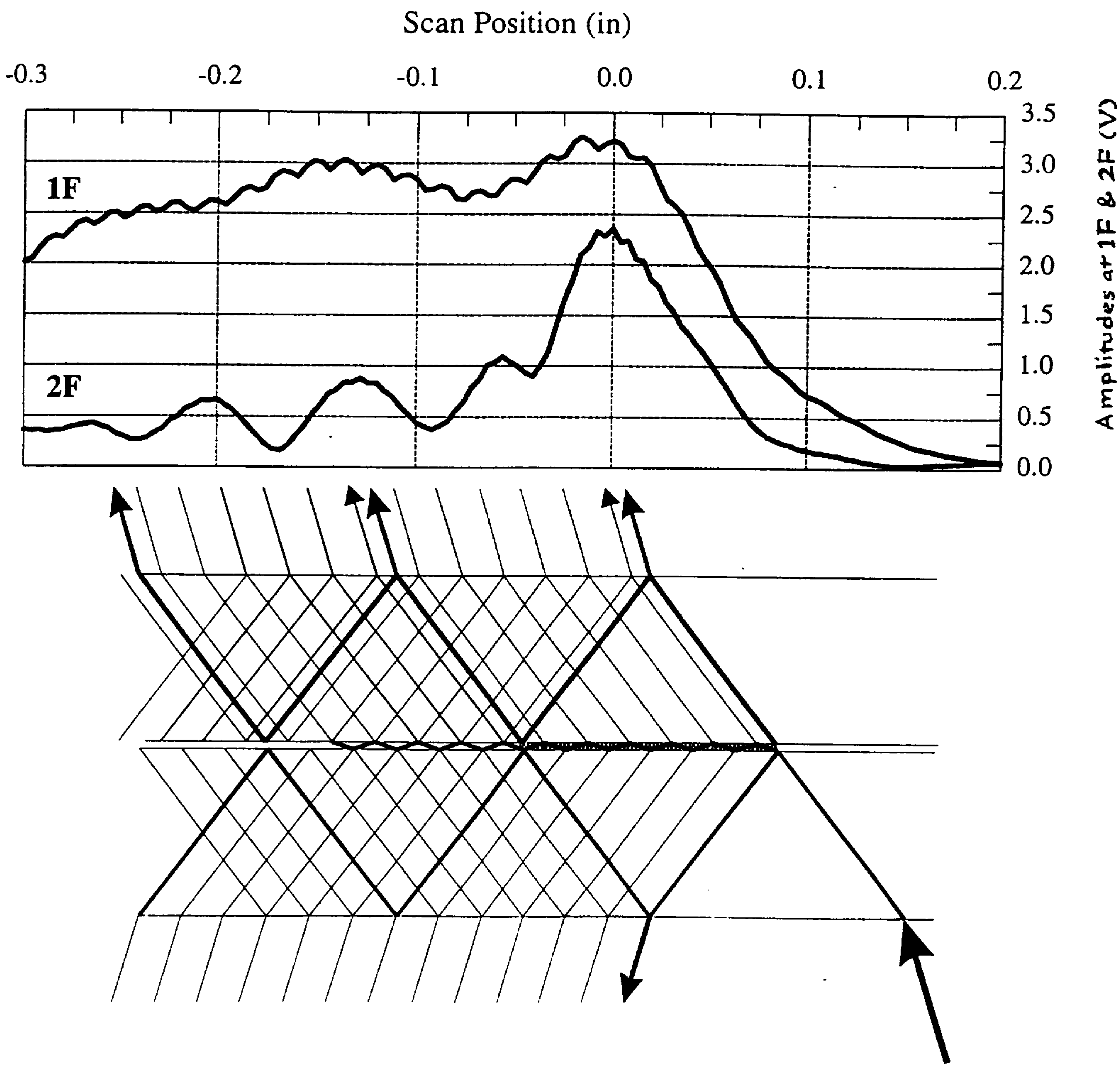
B-Scan of Receiver at 17.5° behind BOEING Sample #19 (good Bond)



B-Scan of Receiver at 17.5° behind BOEING Sample #22 (with Peel Ply)



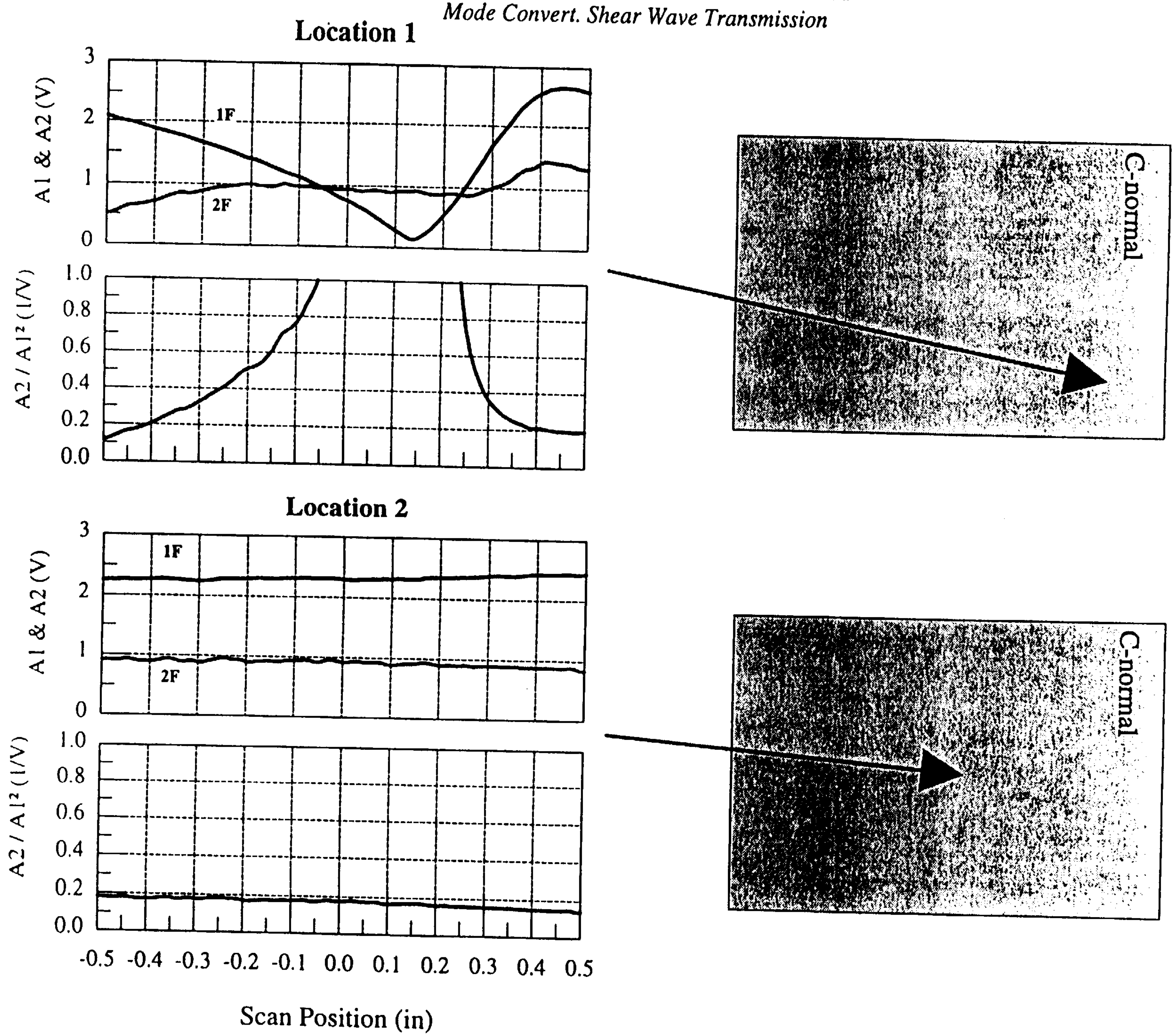
BOE1922C PGW


Scan Position (in)
-0.3
-0.2
-0.1
0.0
0.1
0.2
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
1F
2F
Amplitudes at 1F & 2F (V)

MCSW_D_1 PGW

B-Scan of C-normal for 17.5° and 83 Vpp Drive

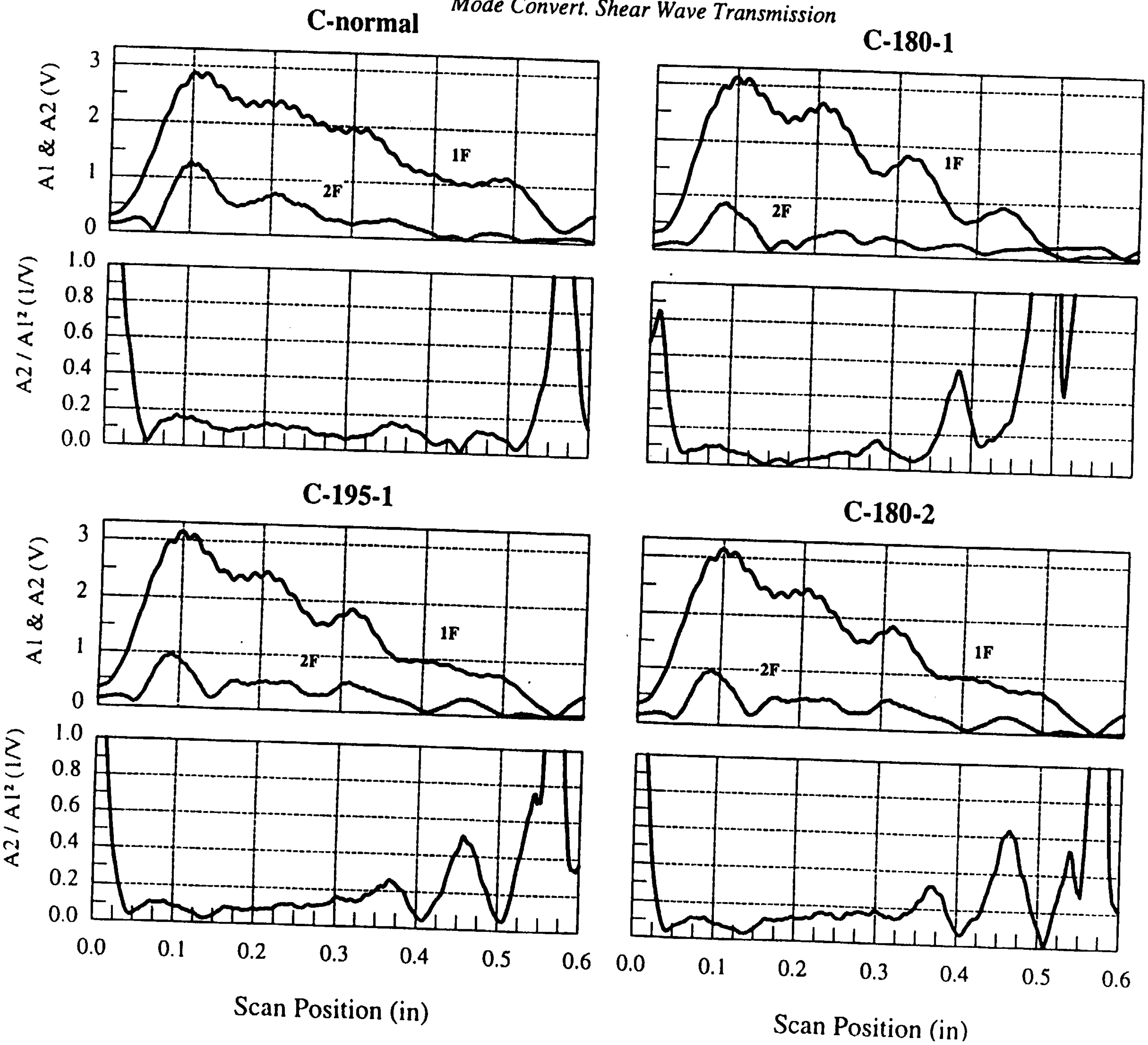
Mode Convert. Shear Wave Transmission



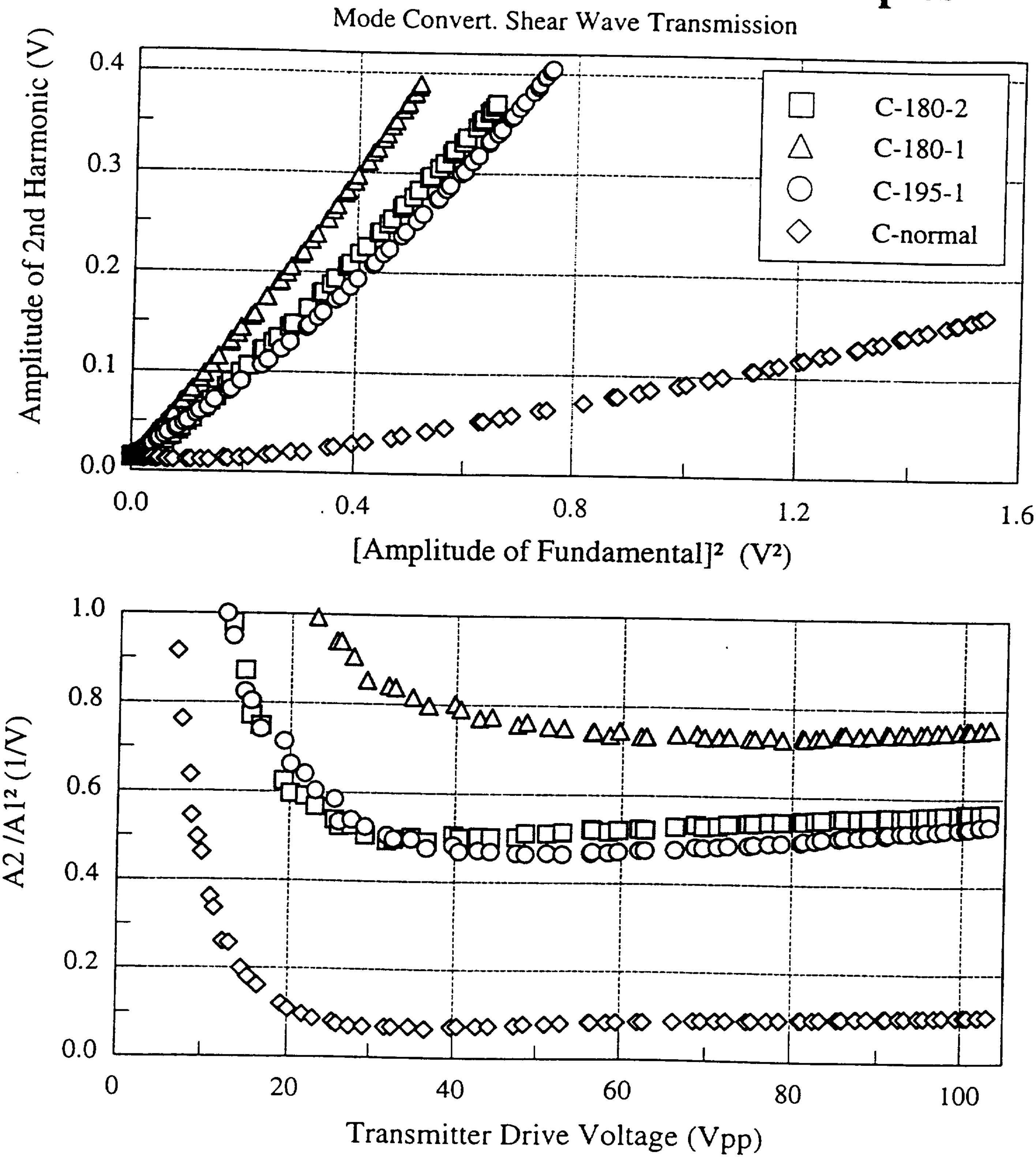
CnormBSC0.PGW

B-Scan of Receiver for 17.5° and 83 Vpp Drive

Mode Convert. Shear Wave Transmission



Comparison of all "CURE" Samples



C_GAV7C1.PGW

Summary

- It has been shown that Measurements of Sample Nonlinearities in Water Immersion are possible if the Power of the injected Fundamental Wave is limited to prevent Interference due to excessive Nonlinear Behavior of the Water.

- Nonlinear Ultrasonic Studies on Adhesive Lap Joint Samples have been performed using Water coupling.

- Lap Joints containing Polyester Peel Plies show Nonlinearities up to four times higher than Ordinary Samples.

- First tests on Samples containing Bond Degradations due to Variations in the Cure Cycle of the Adhesive appear to be more difficult.

Preliminary Attempts to Detect Weakness of Adhesive Bonds

Acousto-elastic Measurements Using Plate Waves

Don Price

CSIRO Telecommunications & Industrial Physics
Sydney, NSW, Australia

Boeing/CSIRO Joint Research Program

NDT of Bonded Structures

Previous work:

- **Delamination of Al-Al bonded joints (and detection of hidden corrosion).**
- **Detection of foreign material inclusions in composite laminates.**
- **Measurement of elastic constants of composite laminates (high temperature ageing).**
- **Measurement of bond strength.**

People involved:

- Barry Martin
- Jill Ogilvy
- Don Price
- Wayne Woodmansee

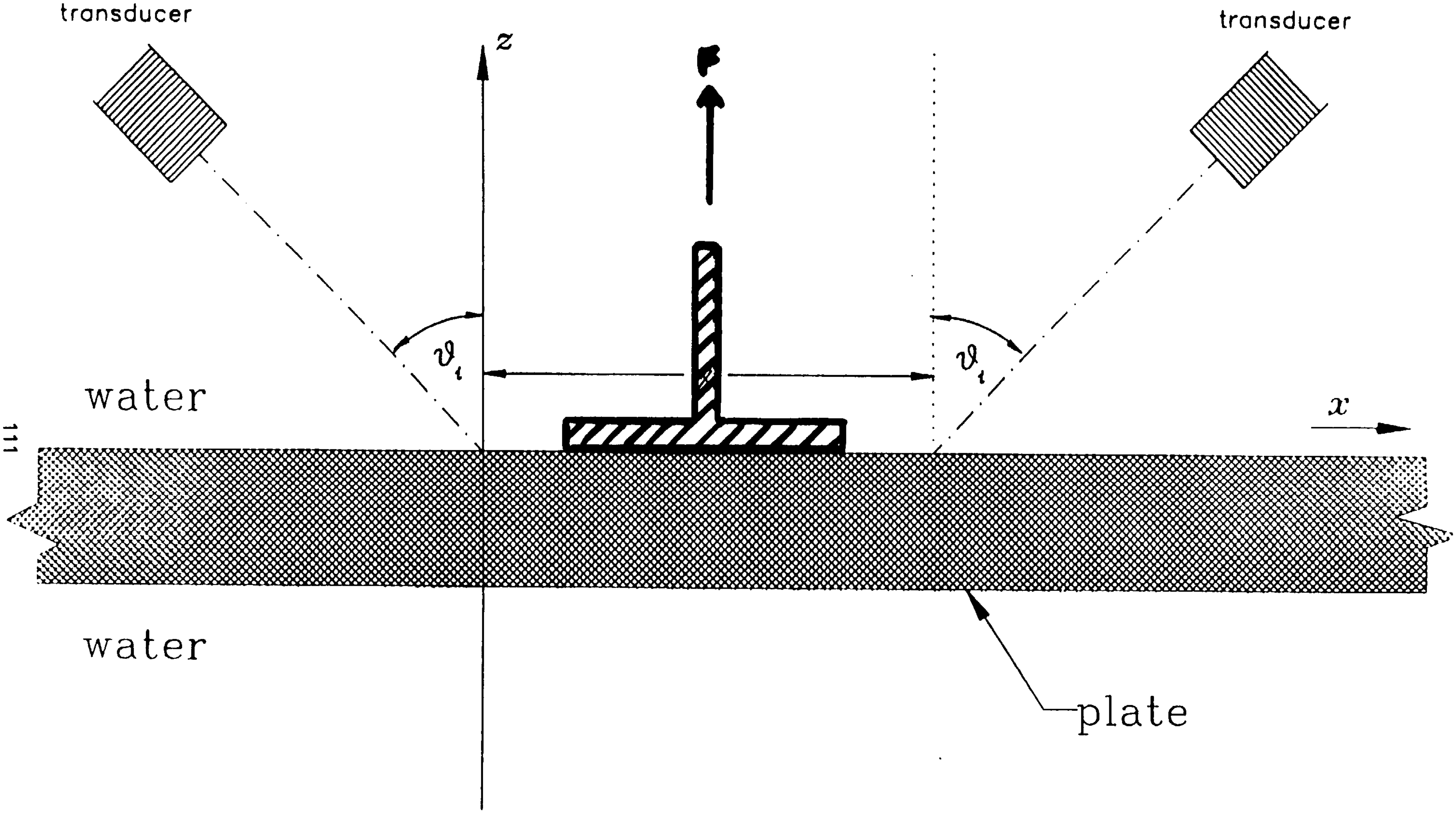


Figure 2.1 : A schematic diagram of the experimental arrangement used for the measurements reported here. The distance x is referred to as the transducer separation.

Sample panels:

(1) Composite substrate & stiffener

- 88 ply carbon fibre/epoxy base plate
- Composite stringer
  (both typical of parts used on 777)
- peel ply removed from one stiffener, left in place on one.

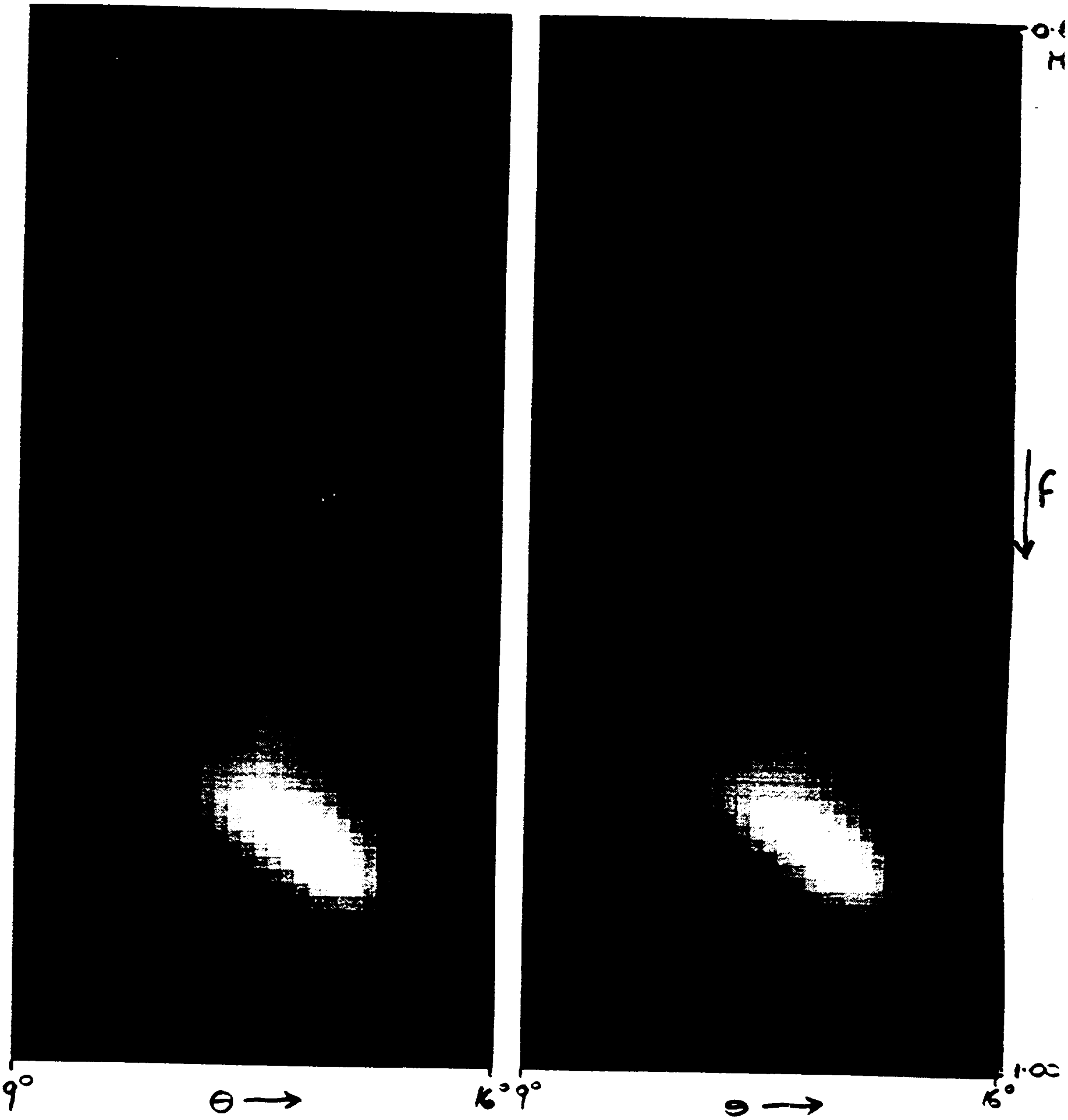
(2) Aluminium baseplate & stiffener

- $\frac{1}{2}$" thick primed Al baseplate
- inverted 'T' Al stiffener
- peel ply included in bond for one stiffener

(3) Aluminium baseplate, composite stiffener

- $\frac{1}{2}$" thick primed Al baseplate (as 2)
- Composite stringer (as in 1)
- room temp. cure paste adhesive.

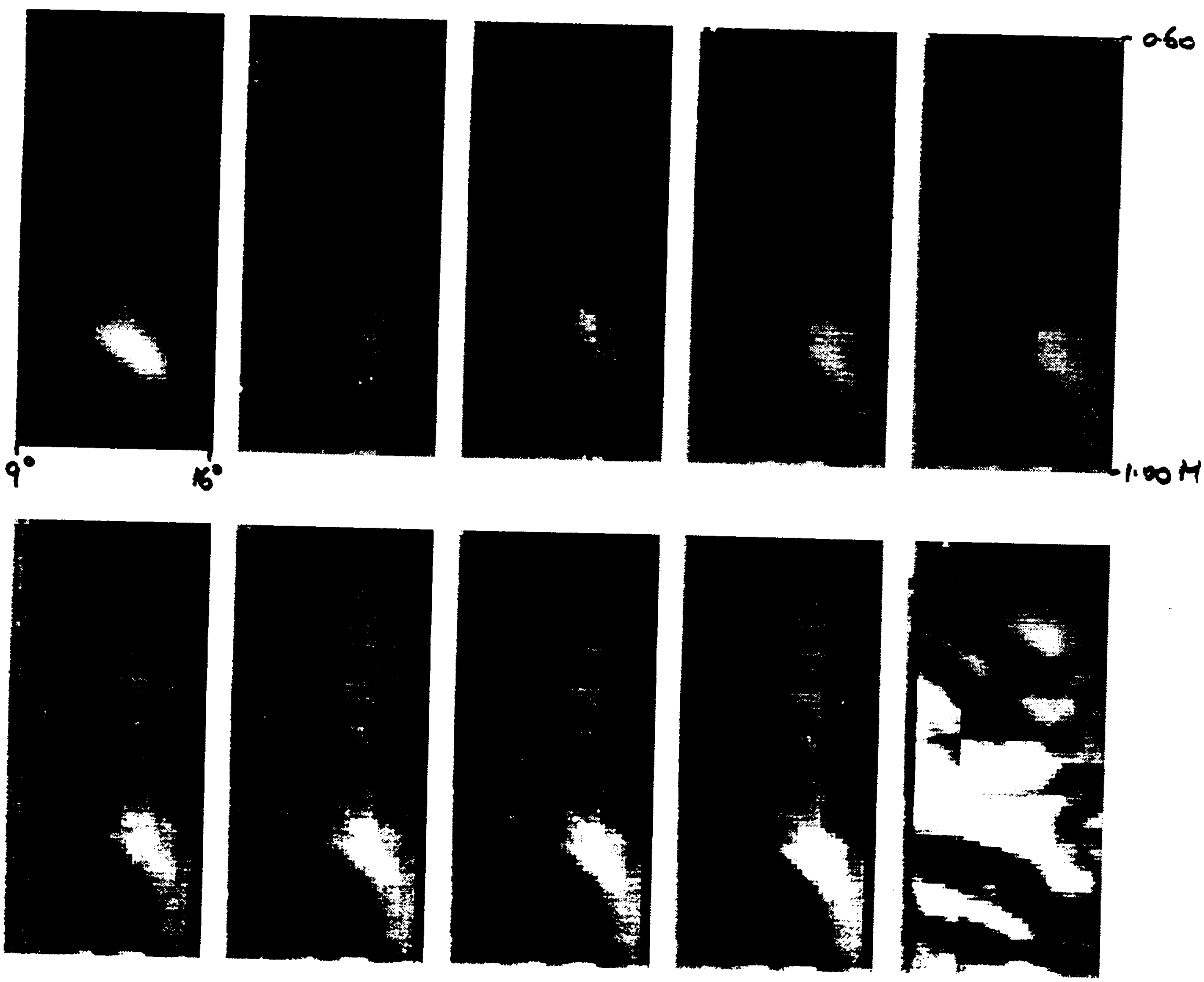
Received Signal Amplitude
(Zero applied stress)



Stiffener #R
well-bonded

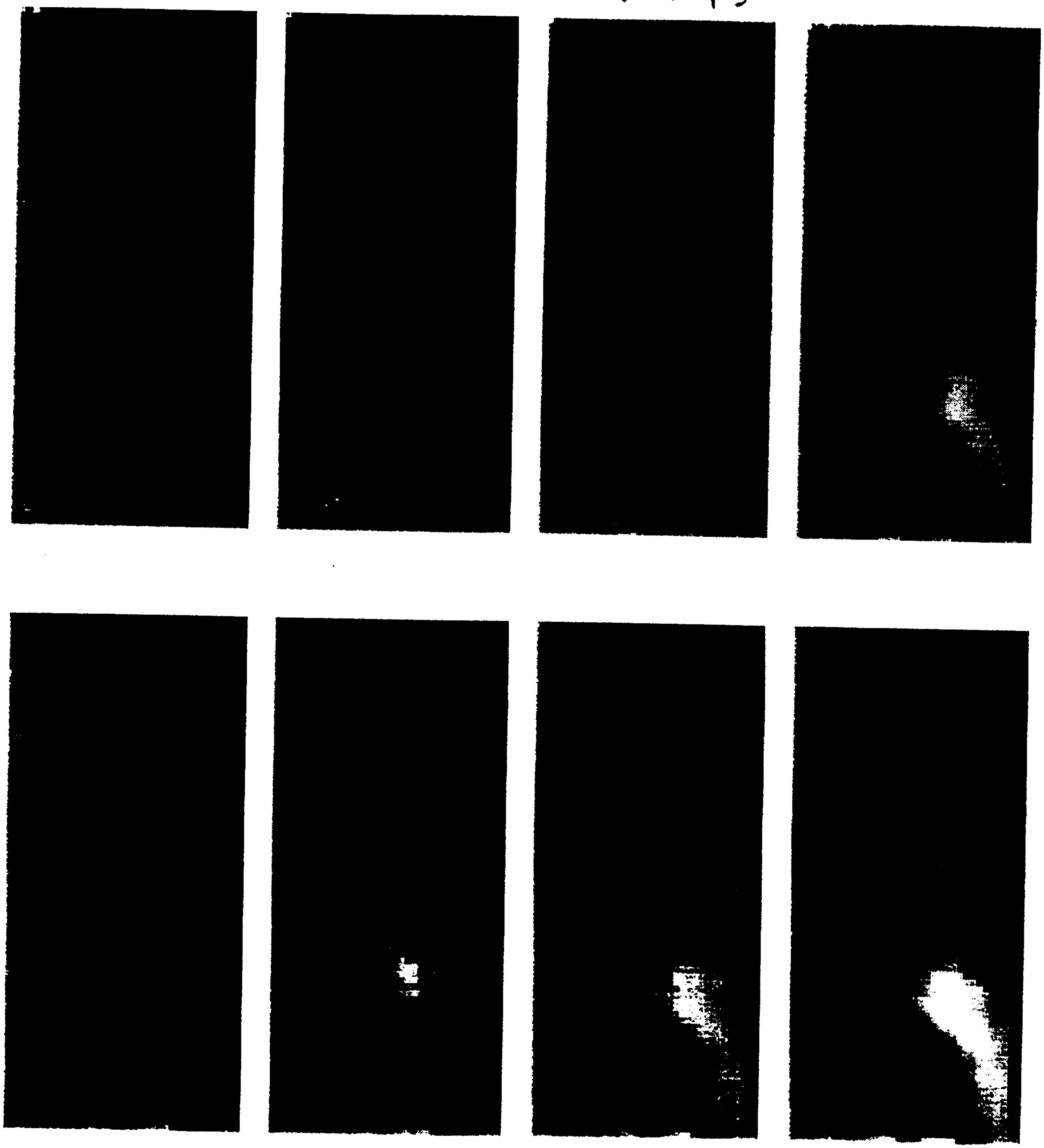
Stiffener #F
Peel ply

Change in received signal amplitude
with increasing stress
Peel ply stiffener (#P)



Change in received signal amplitude with increasing tensile stress

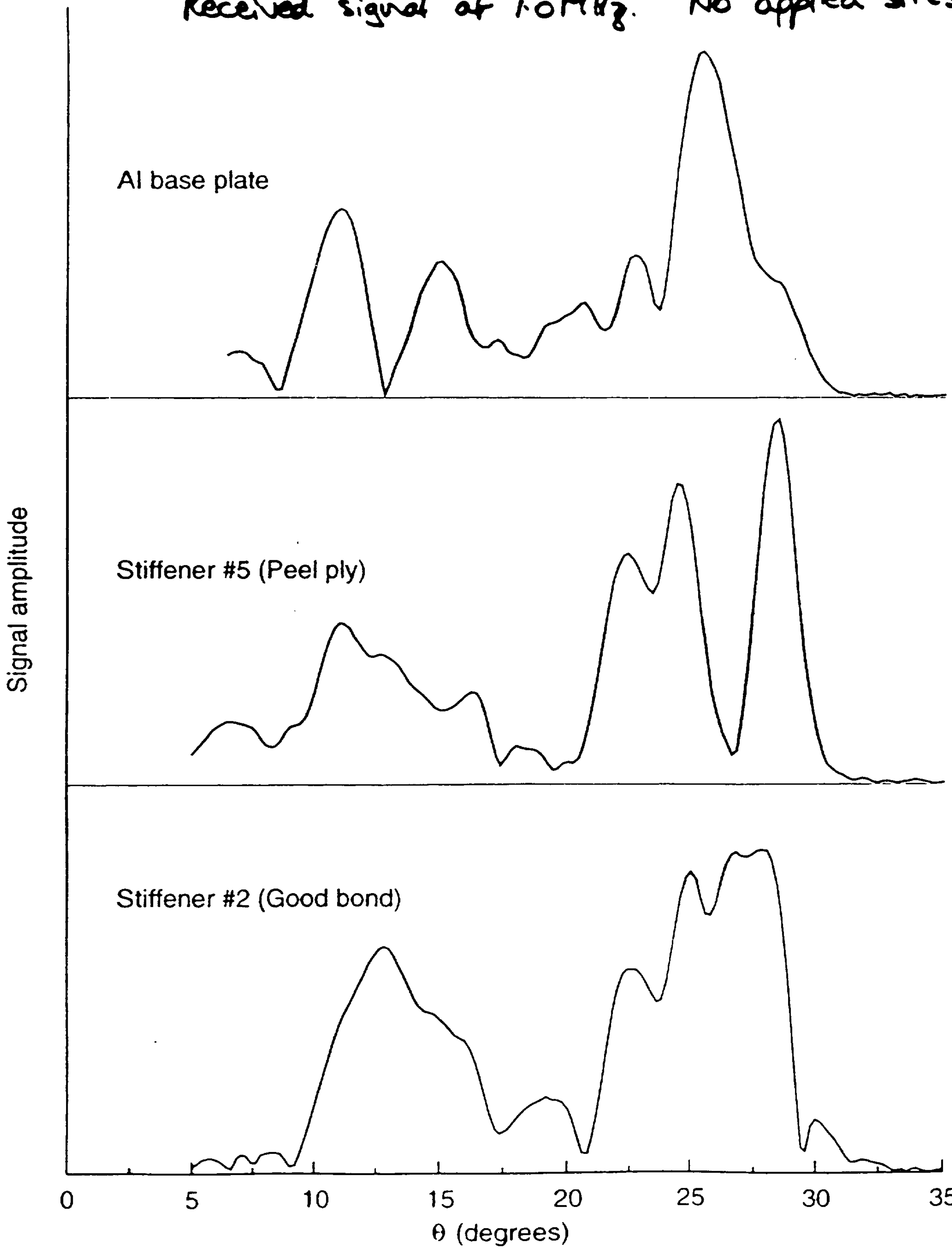
Well-bonded stiffener (top)



Peel ply stiffener (lower)

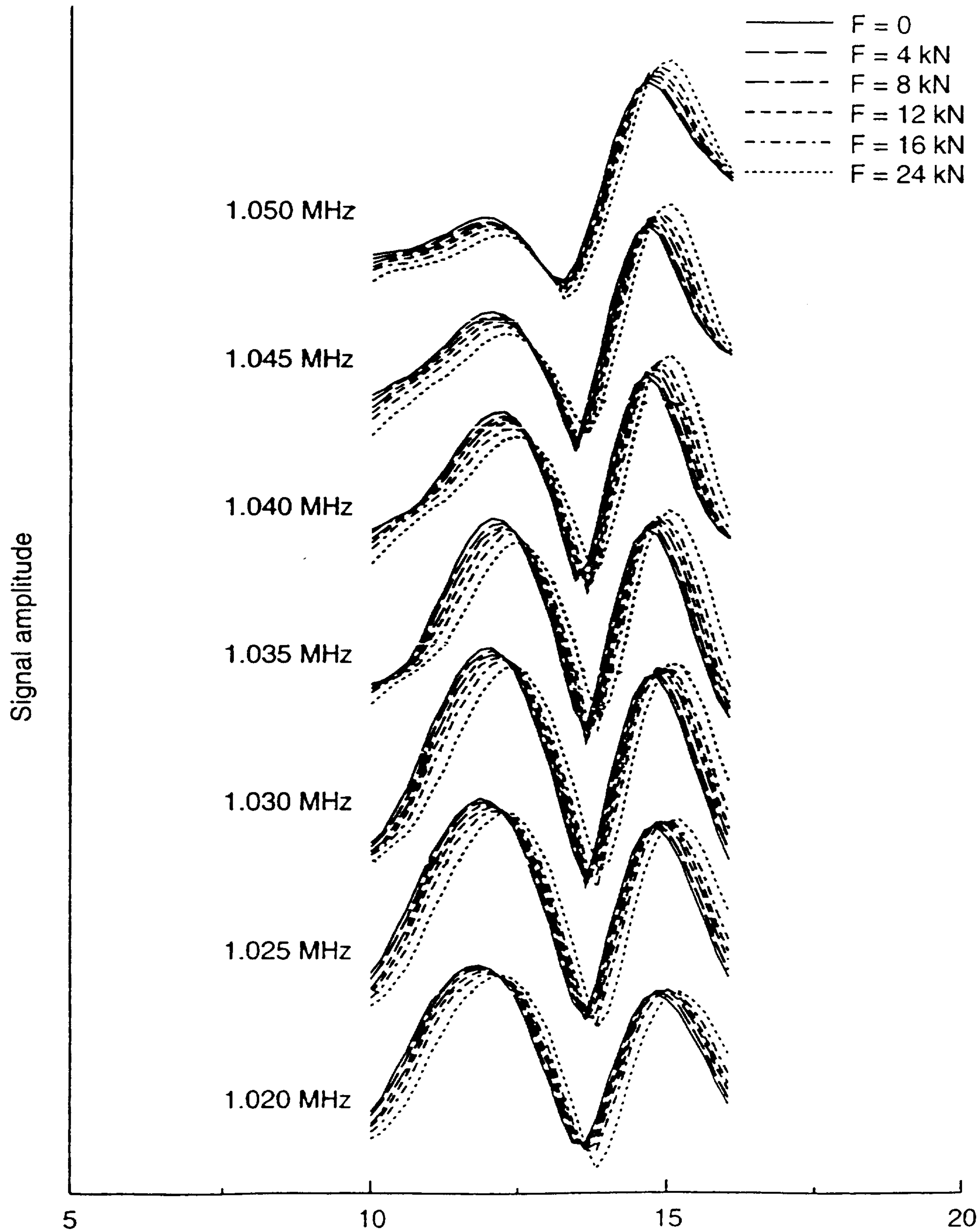
Sample no.6 : Al baseplate
Al stiffeners

Received signal at 1.0MHz. No applied stress.



Sample no. 2 : Al - Al

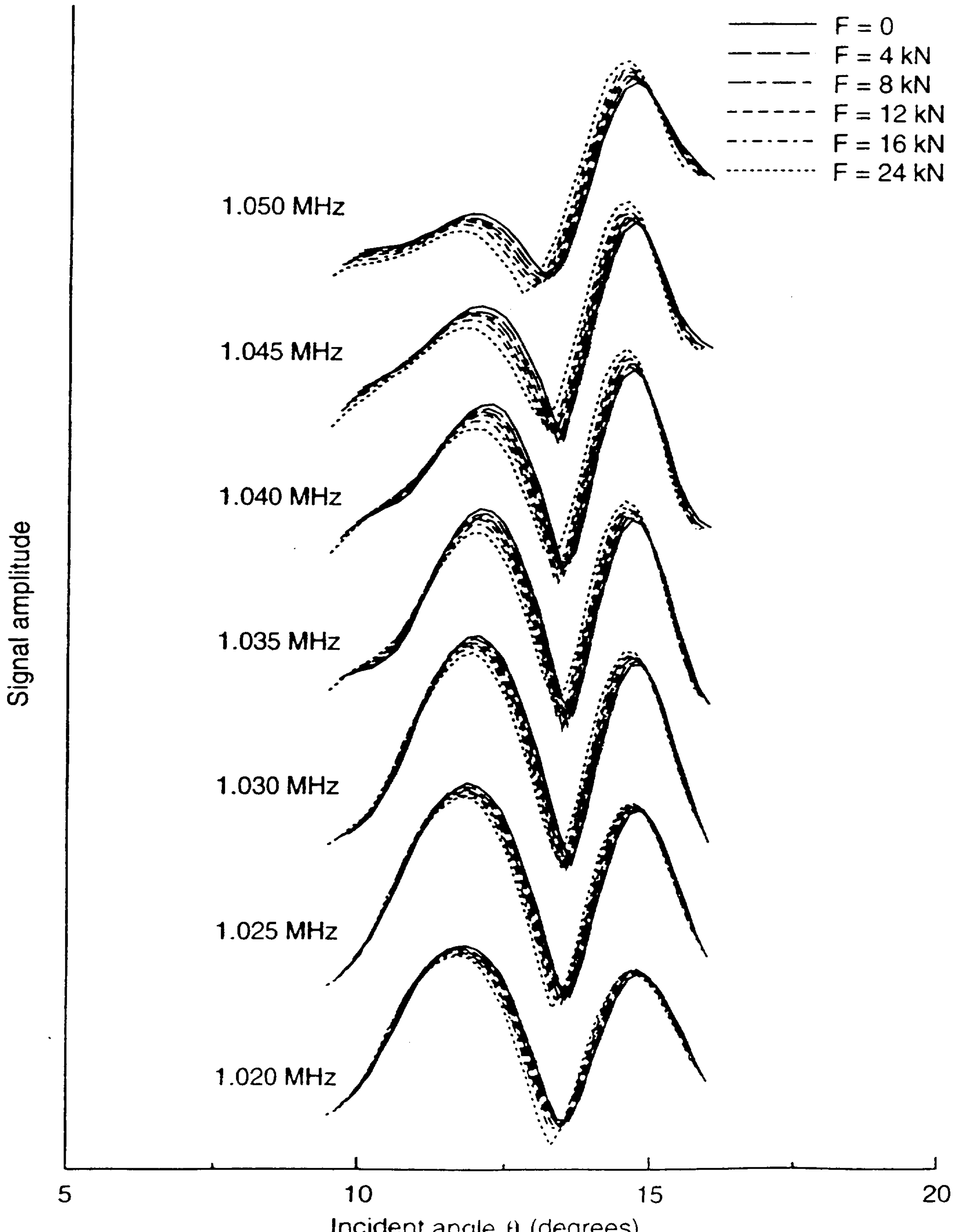
Peel ply stiffener #5



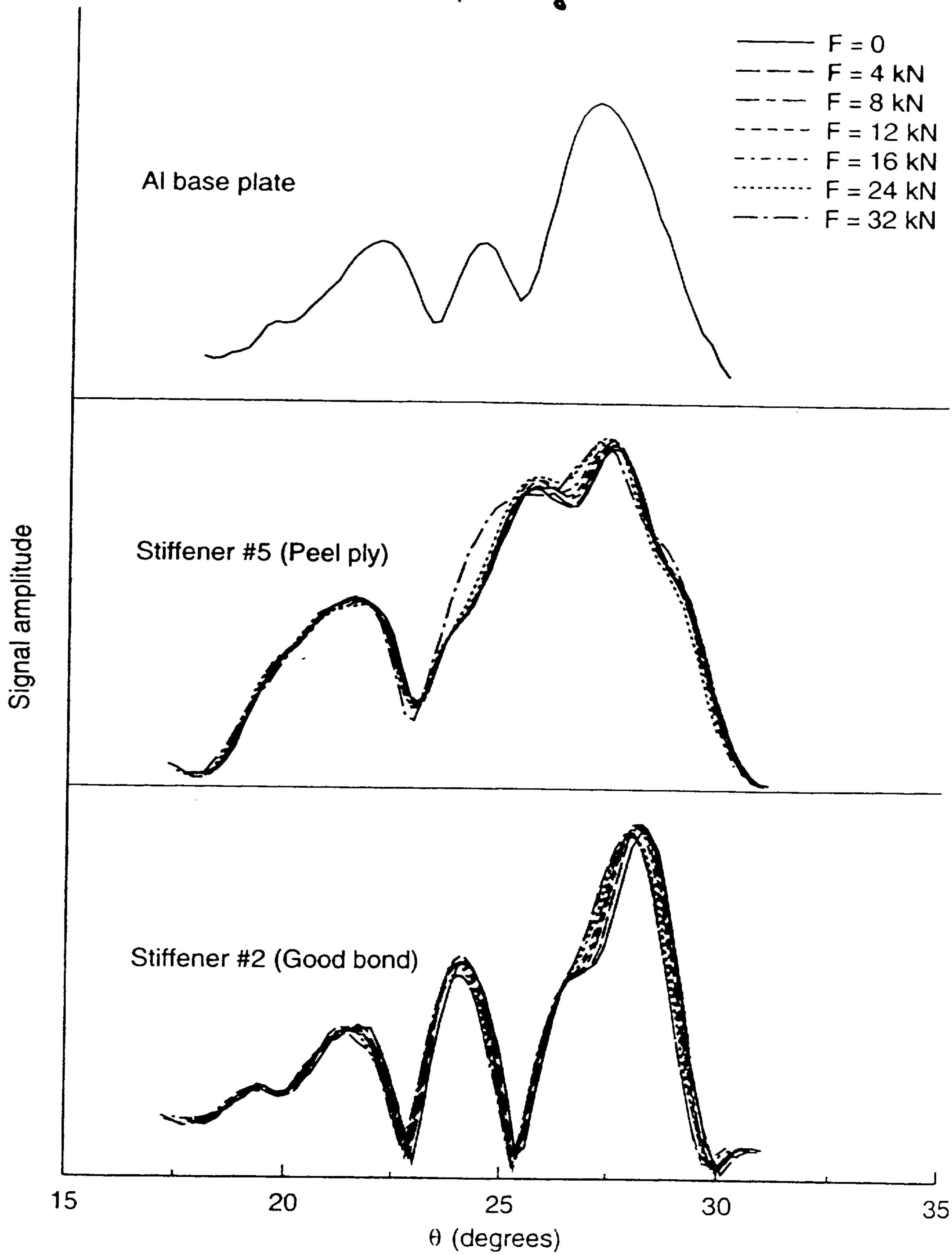
Sample no 2: [illegible]

Peel ply stiffener #5

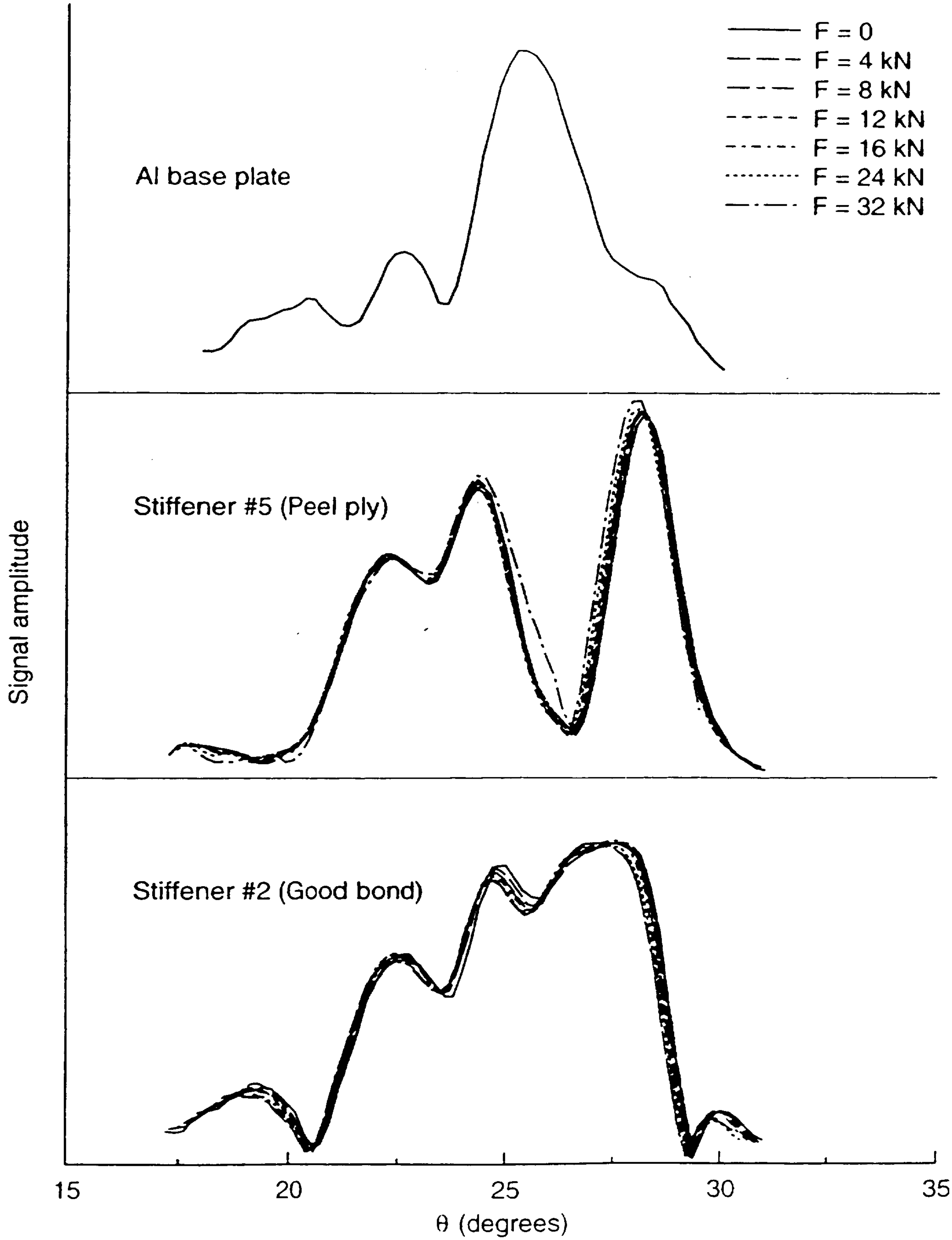
With correction for uniform bending



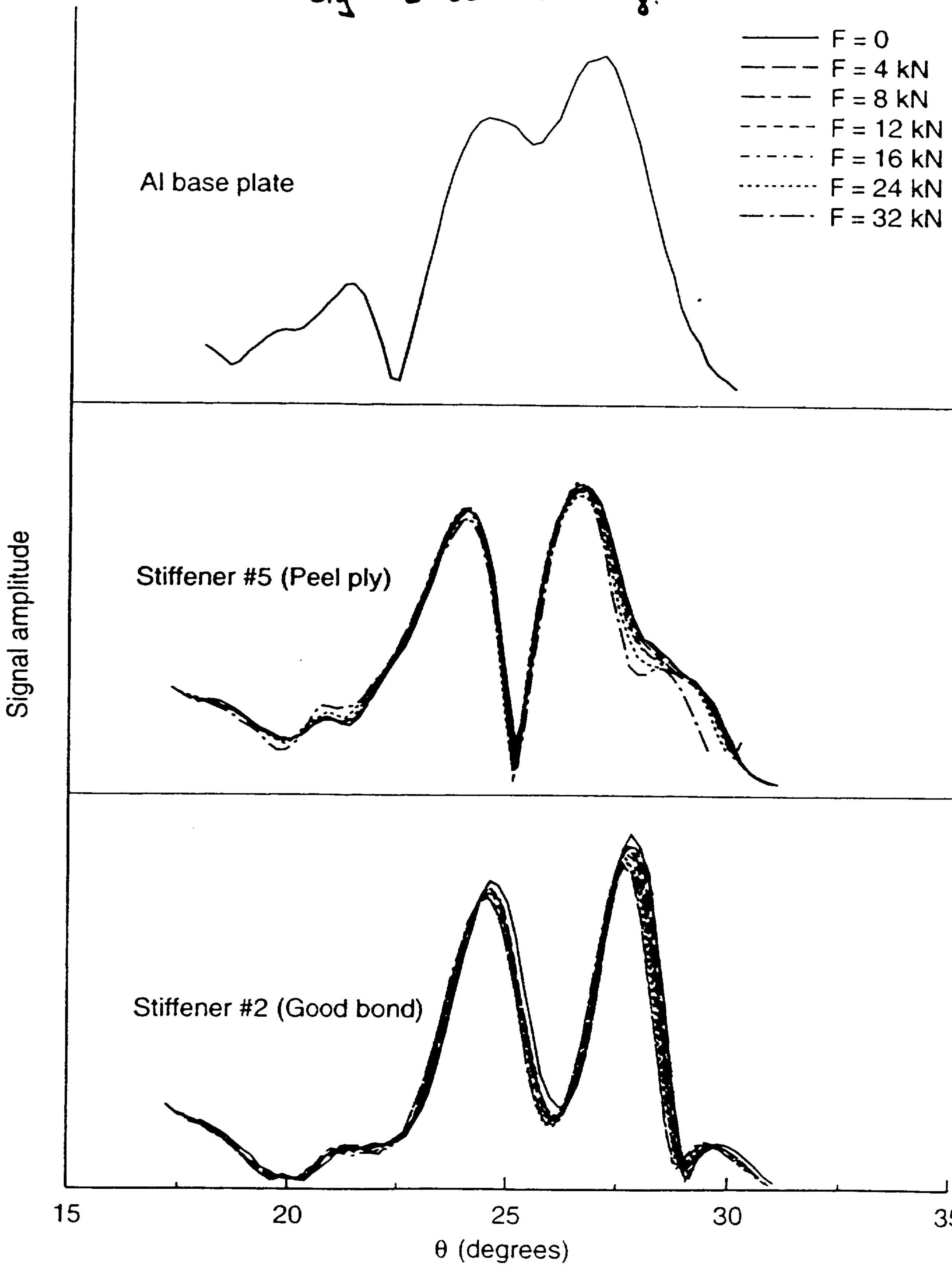
Signals at 0.90 MHz



Sample no. 2
Signals at 1.00 MHz

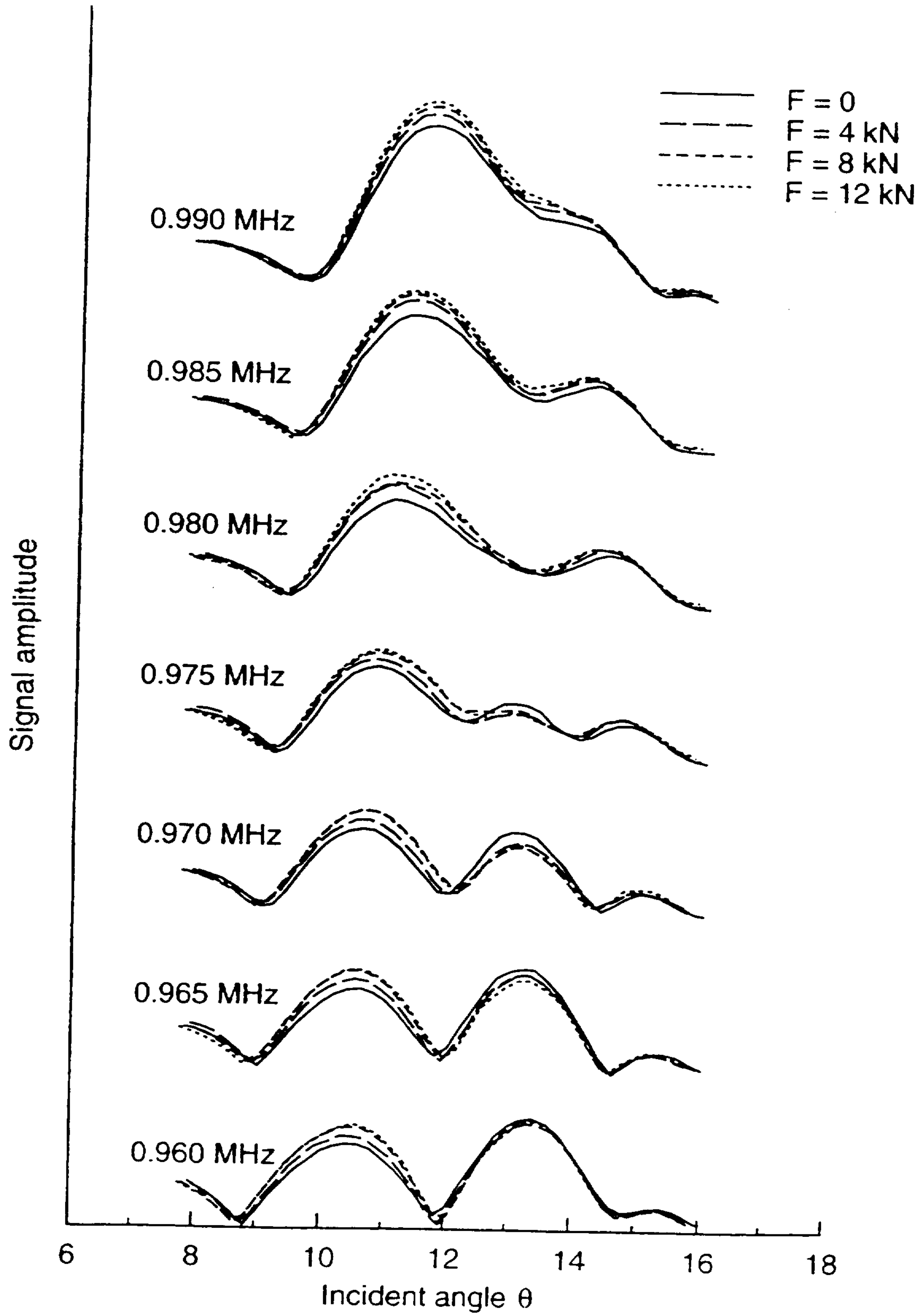


Sample no. 2
Signals at 1.10 MHz.

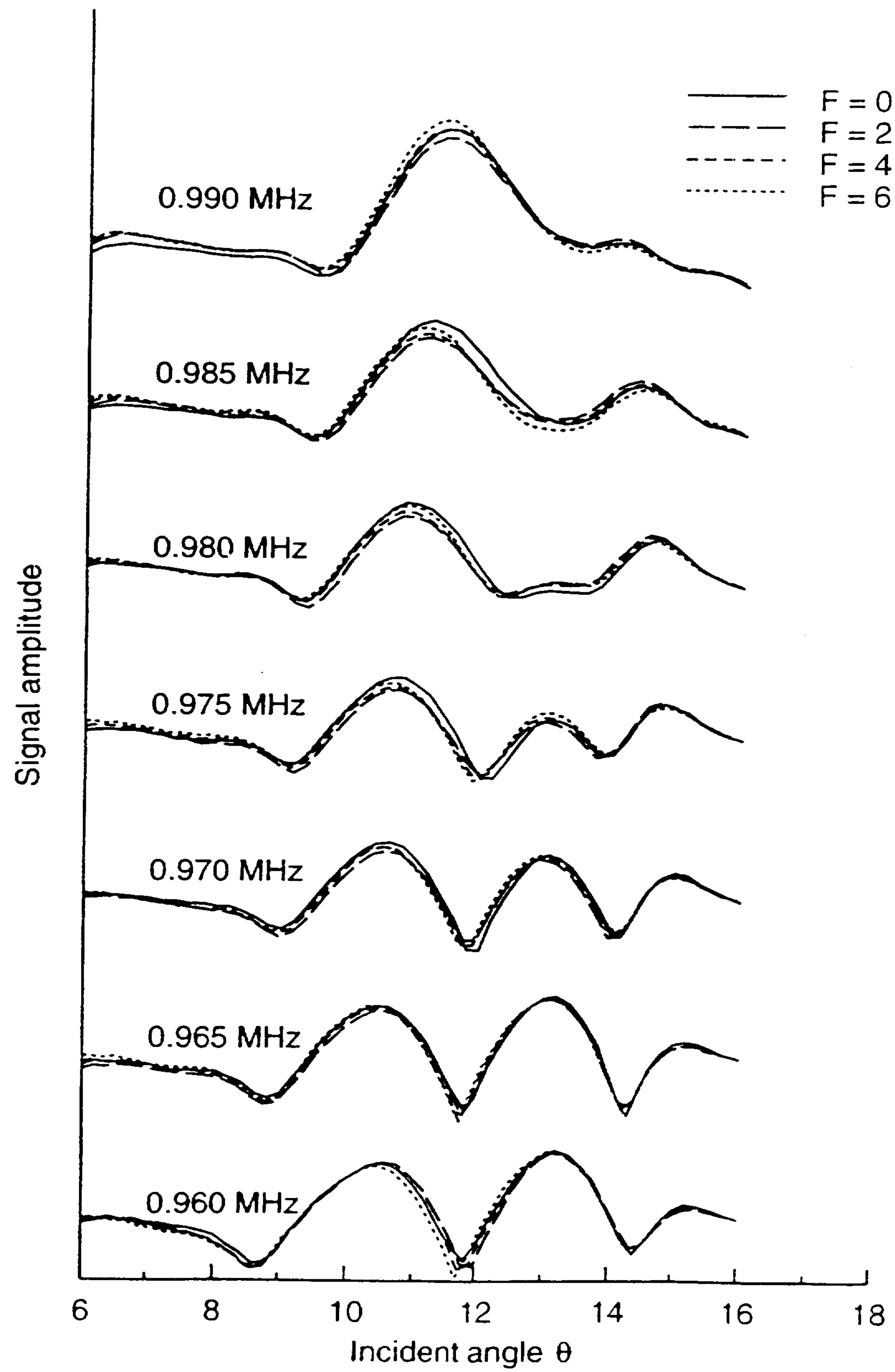


Sample no.3 : Al/Comp.

Stiffener #1 : no ped ply

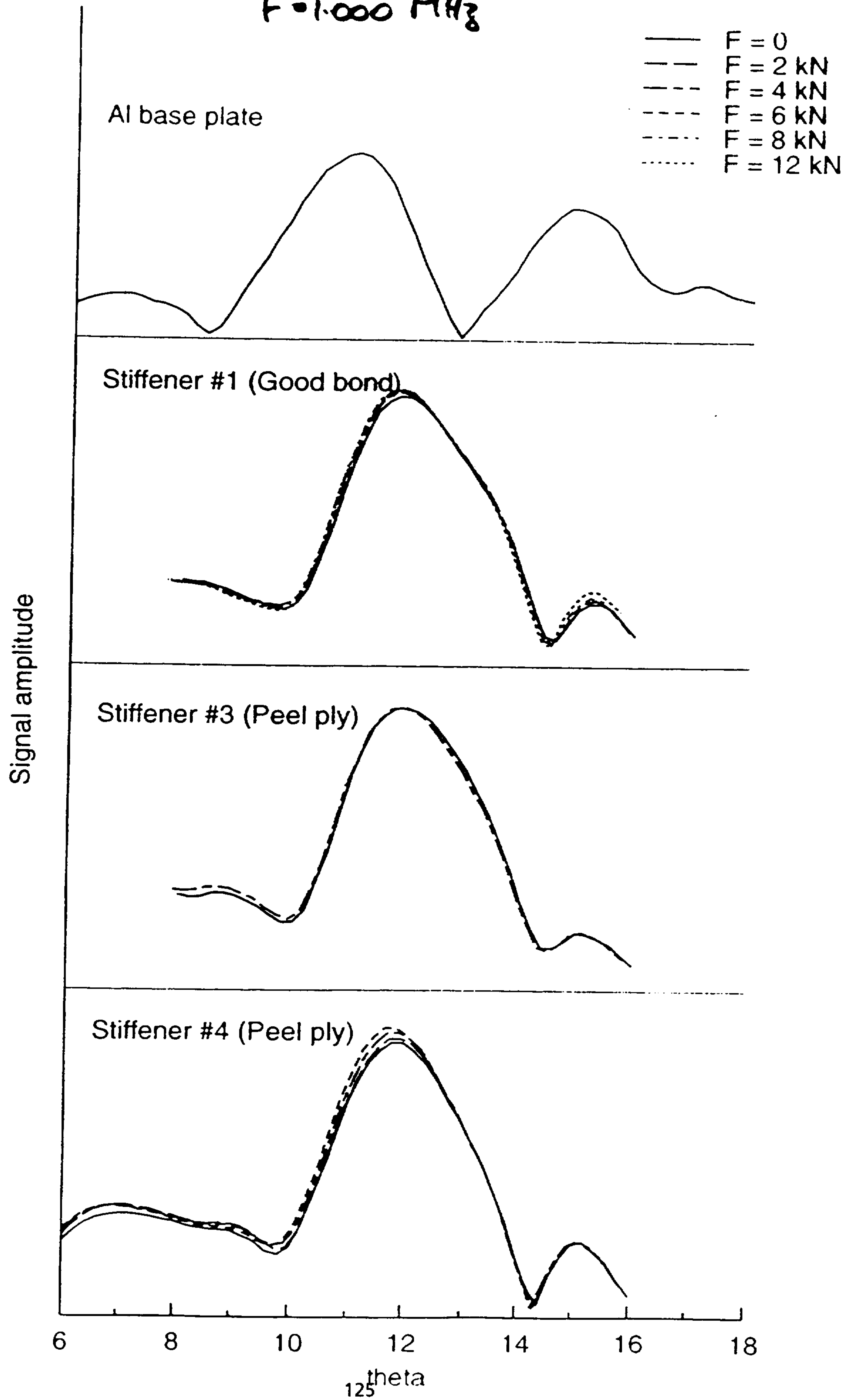


Stiffener #4: peel ply.

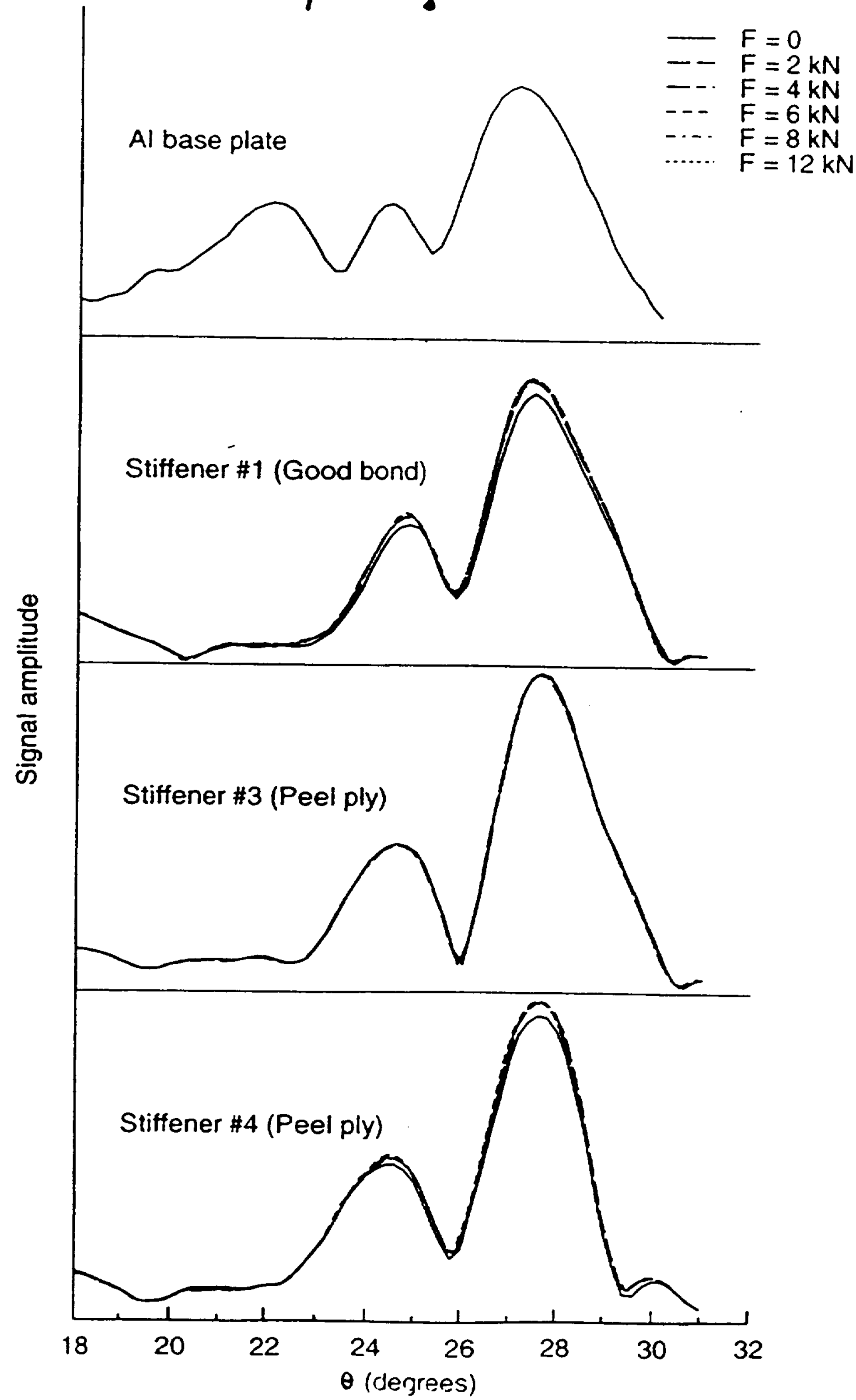


Sample no. 5. AL/Comp

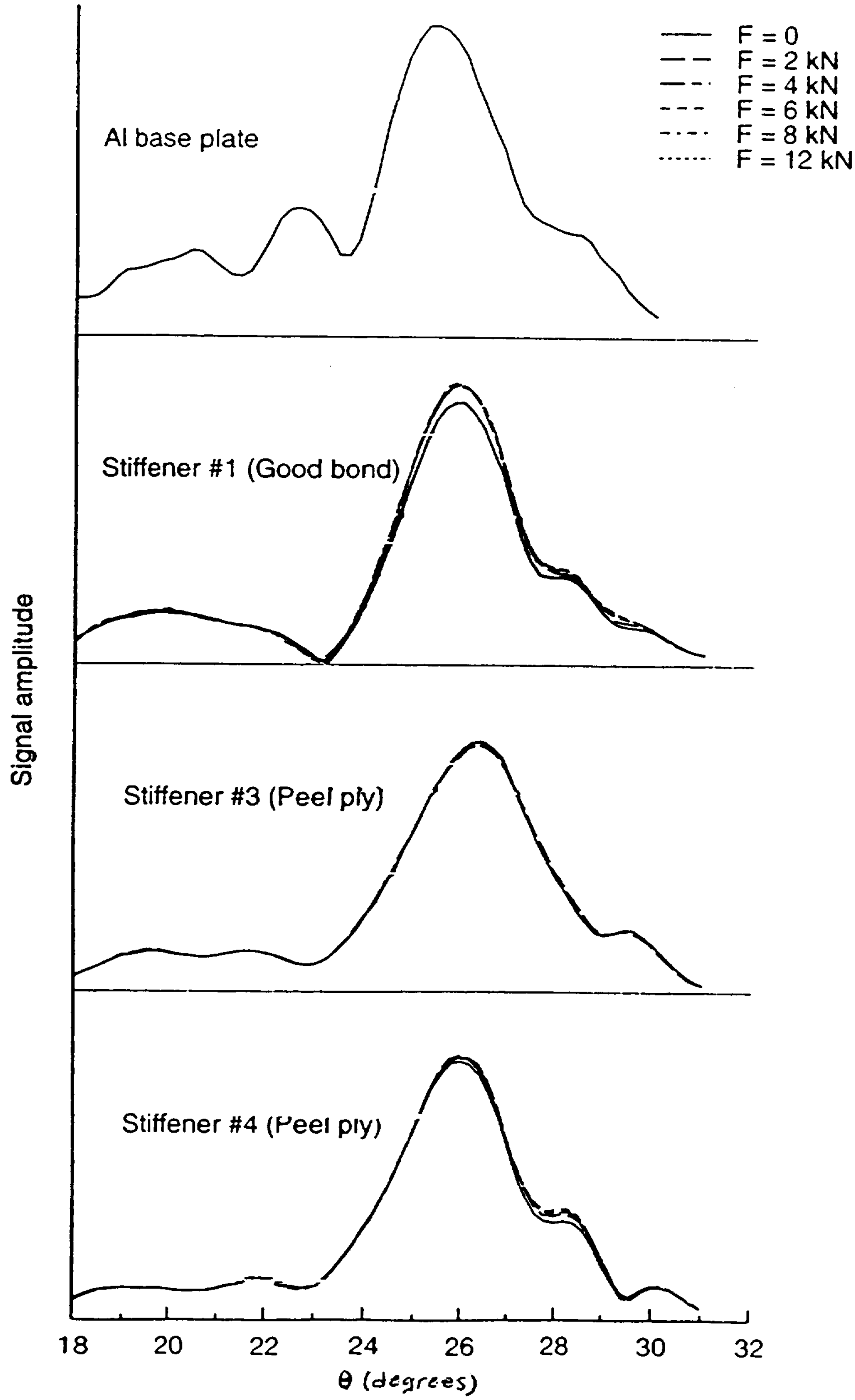
f = 1.000 MHz



Sample no.3: Al/comp
f = 0.900 MHz



Sample no.3: Al/comp
f = 1.000 MHz



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13. ABSTRACT (*Maximum 200 words*)
Quantitative adhesive bond strength measurement has been an issue for over thirty years. Utilization of nonlinear ultrasonic nondestructive evaluation methods has shown more effectiveness than linear methods on adhesive bond analysis, resulting in an increased sensitivity to changes in bondline conditions. Correlation to changes in higher order material properties due to microstructural changes using nonlinear ultrasonics has been shown and could relate to bond strength. Nonlinear ultrasonic energy is an order of magnitude more sensitive than linear ultrasound to these material parameter changes and to acoustic velocity changes caused by the acoustoeleastic effect when a bond is prestressed. This increased sensitivity will assist in getting closer to quantitative measurement of adhesive bond strength. Signal correlations between non-linear ultrasonic measurements and initialization of bond failures have been successfully measured. This paper reviews nonlinear bond strength research efforts presented by university and industry experts at the First Annual Symposium for Nondestructive Evaluation of Bond Strength organized by the NDE Sciences Branch at NASA Langley in November 1997.

14. SUBJECT TERMS
Nonlinearity, ultrasonics, fatigue, residual bond strength, adhesive bond, strength modulus, nonlinear parameter, PZT, harmonics, bond cure

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